Supporting Information

C—ON bond homolysis of Alkoxyamines. Part 11: Activation of the nitroxyl fragment.

Gérard Audran,[a]* Paul Brémond,[a]* Sylvain. R. A. Marque,[a,b]* and Toshihide Yamasaki[a]

[a] Aix-Marseille Université, CNRS, ICR, UMR 7273, case 551, Avenue Escadrille Normandie-Niemen, 13397 Marseille Cedex 20 France.

E-mail: sylvain.marque@univ-amu.fr, g.audran@univ-amu.fr, paul.bremond@univ-amu.fr

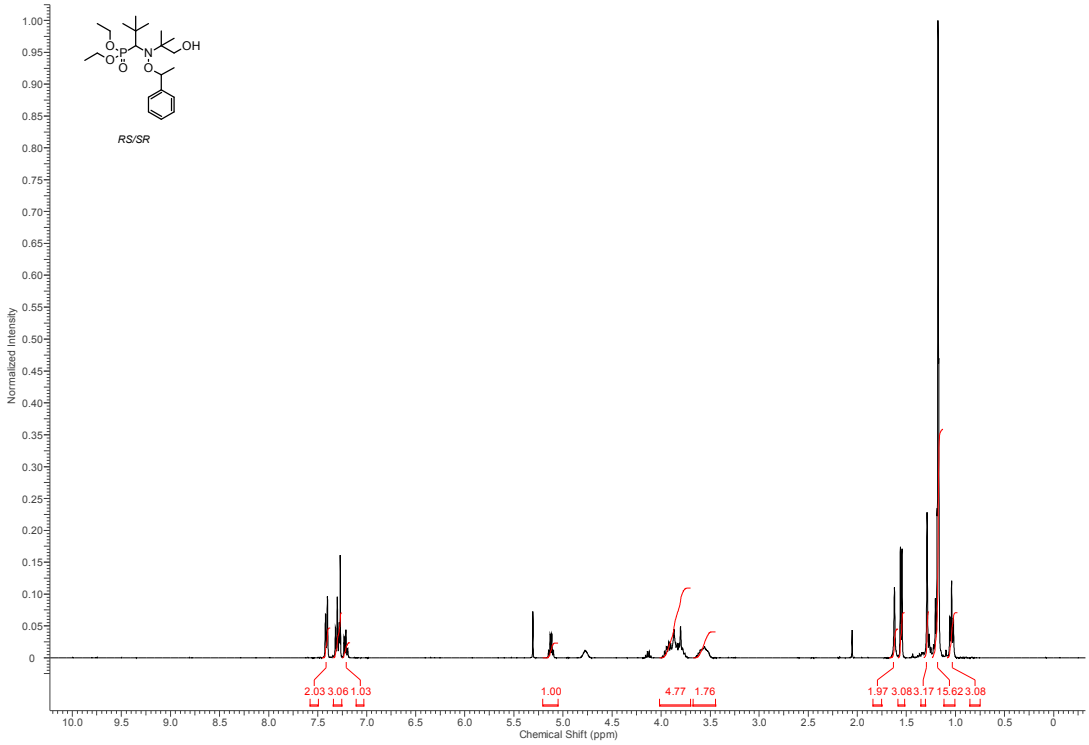
[b] N. N. Vorozhtsov Novosibirsk Institute of Organic Chemistry SB RAS, Pr. Lavrentjeva 9, 630090 Novosibirsk, Russia.

1.	**^{1}H, ^{13}C, ^{31}P NMR spectra of compounds 2-5**	**2**
2.	**Kinetic Measurements**	**18**
3.	**Intramolecular H-bonding**	**18**
4.	**pKa Measurements**	**19**
5.	**X-ray Structures**	**20**
	- (*RR/SS*)-2	**20**
	- (*RS/SR*)-2	**49**
	- (*RR/SS*)-4	**66**
	- (*RS/SR*)-4	**109**
6.	**Reference**	

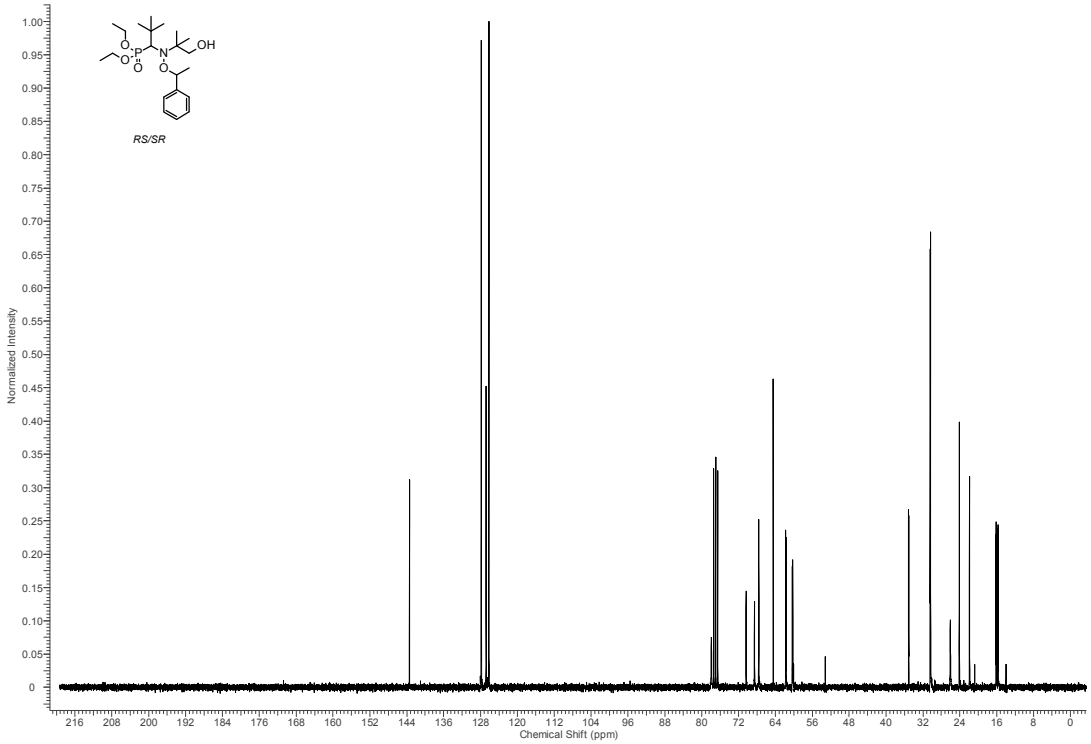
^{1}H, ^{13}C and ^{31}P NMR spectra of compounds 2-5

RS/SR-2:

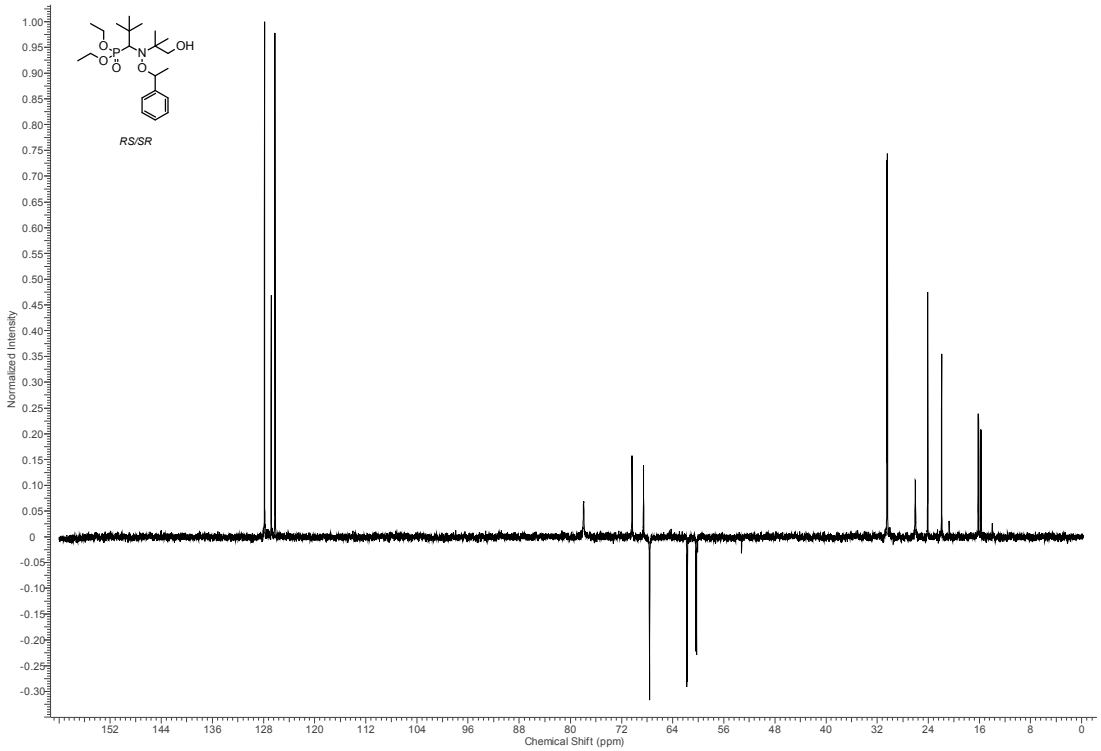
^{1}H-NMR (400 MHz, $CDCl_3$):



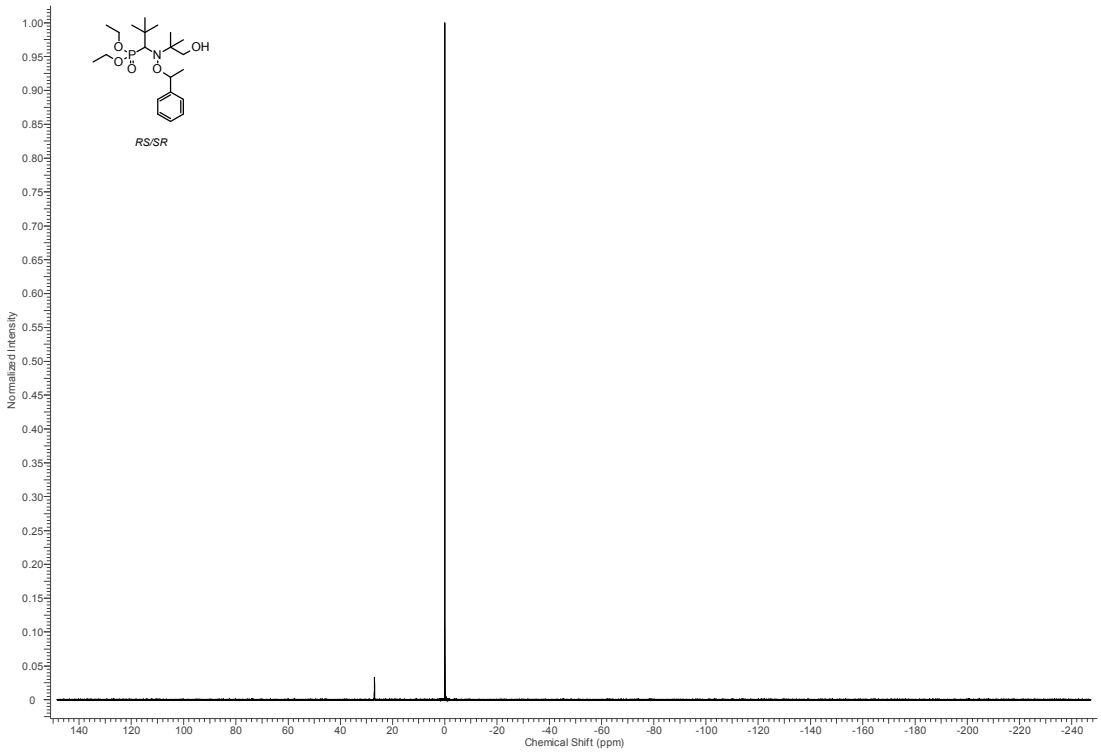
^{13}C{^{1}H}-NMR (75 MHz, $CDCl_3$):



DEPT135 (75 MHz, $CDCl_3$):

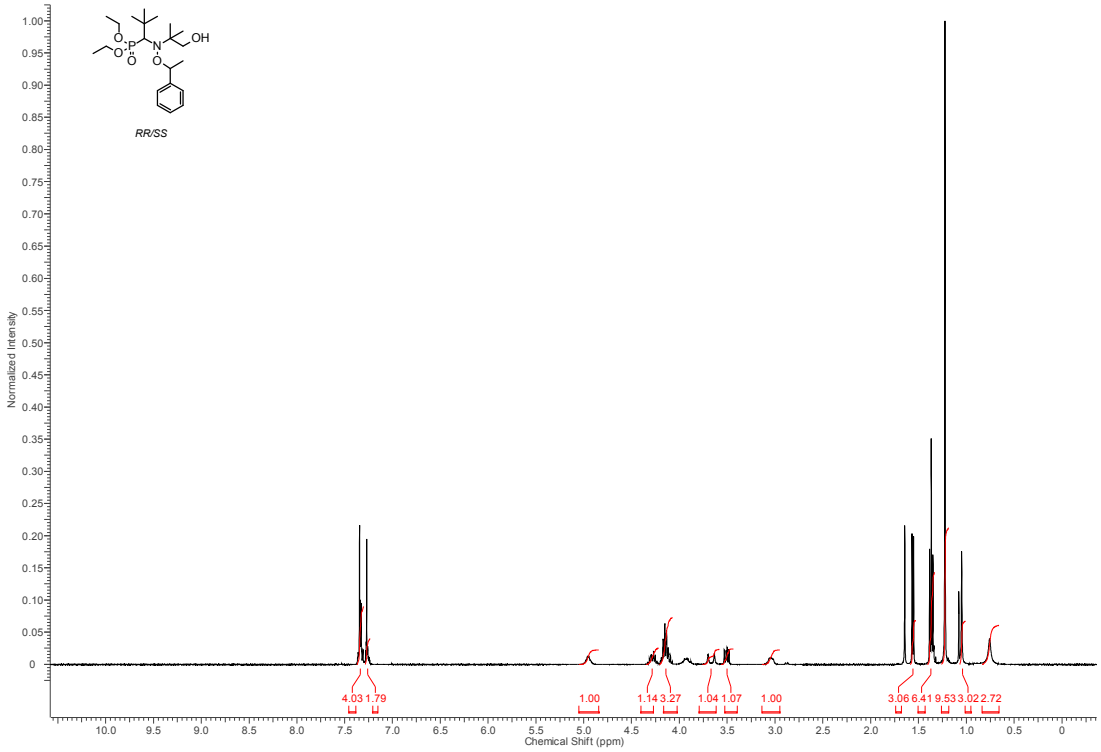


$^{31}P\{^{1}H\}$-NMR (162 MHz, $CDCl_3$):

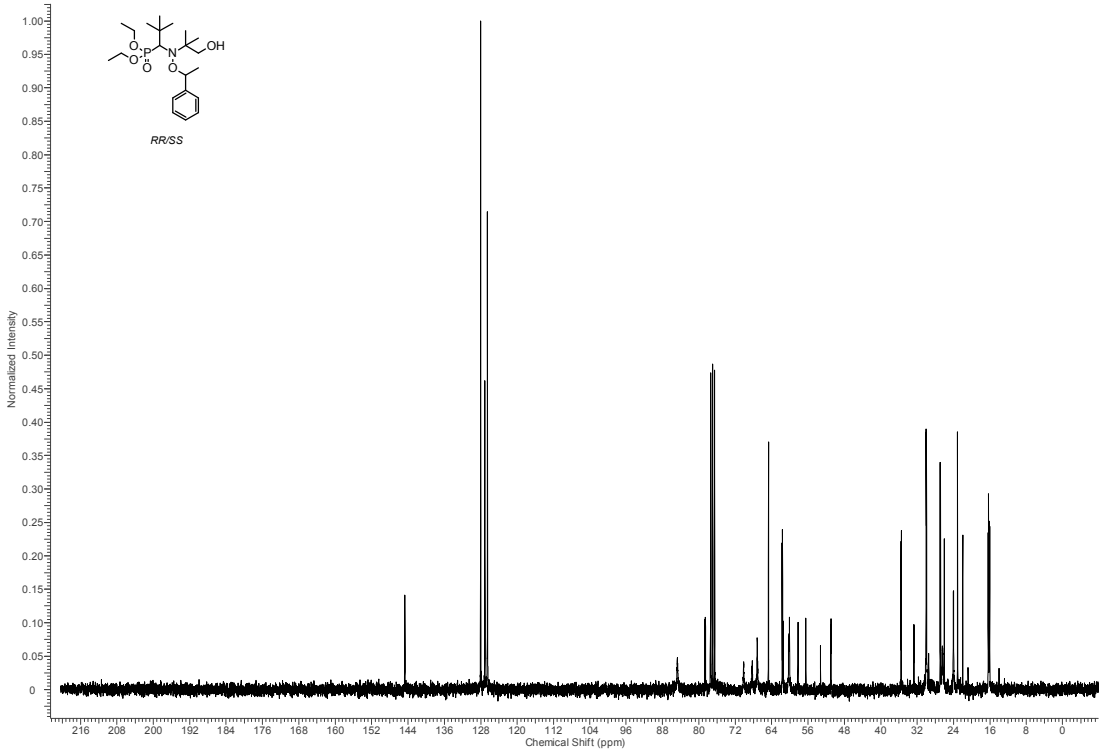


***RR/SS*-2:**

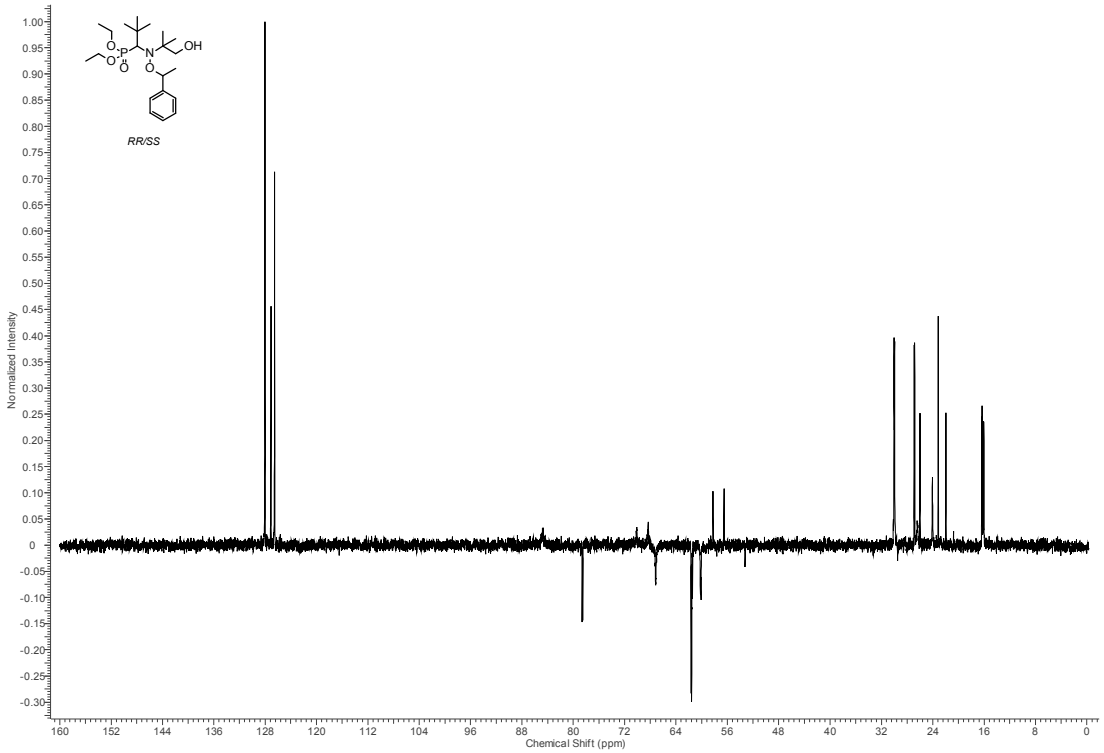
^{1}H-NMR (400 MHz, $CDCl_3$):



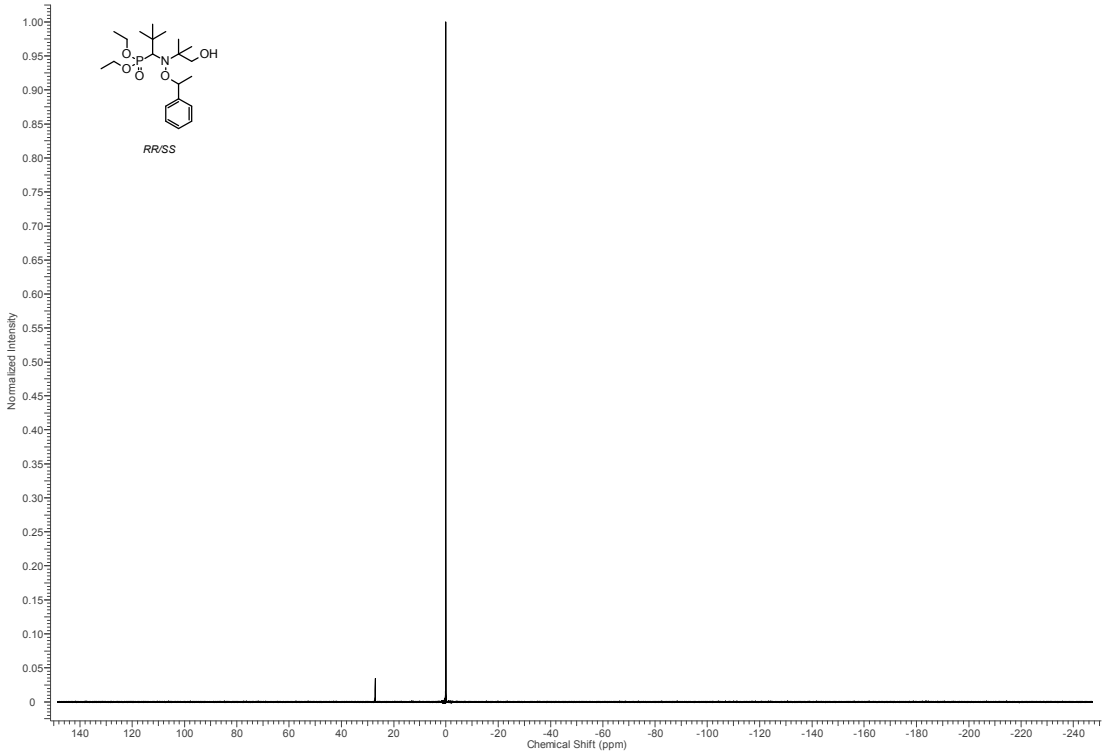
^{13}C{^{1}H}-NMR (75 MHz, $CDCl_3$):



DEPT135 (75 MHz, $CDCl_3$):

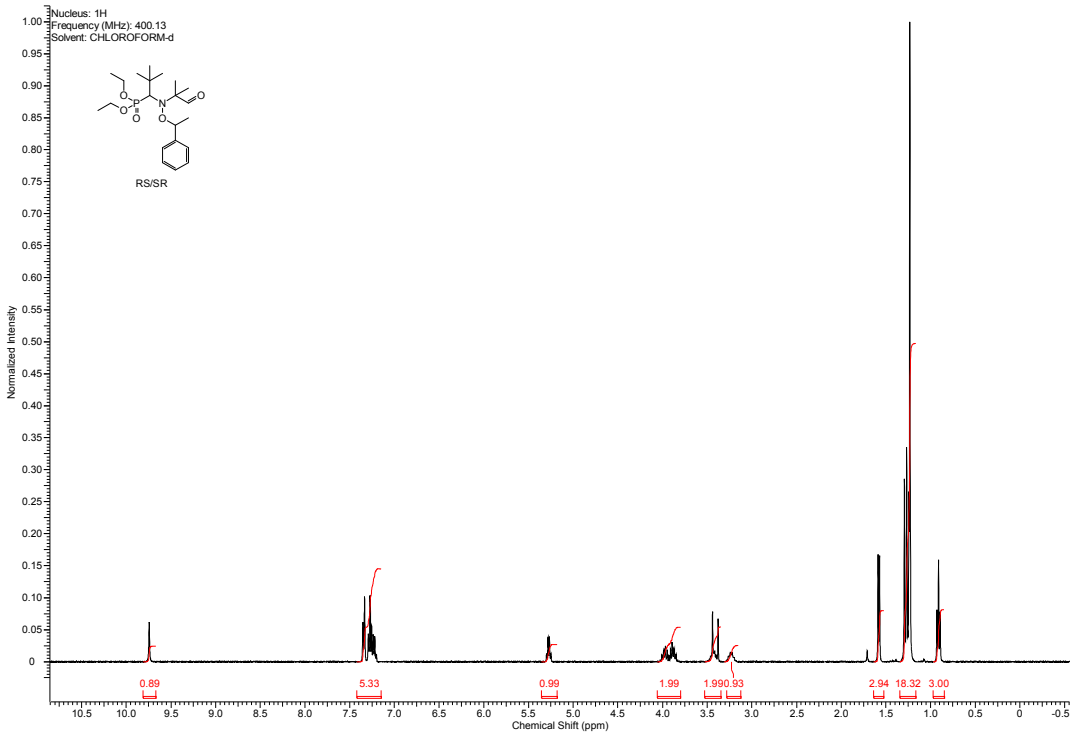


$^{31}P\{^{1}H\}$-NMR (162 MHz, $CDCl_3$):

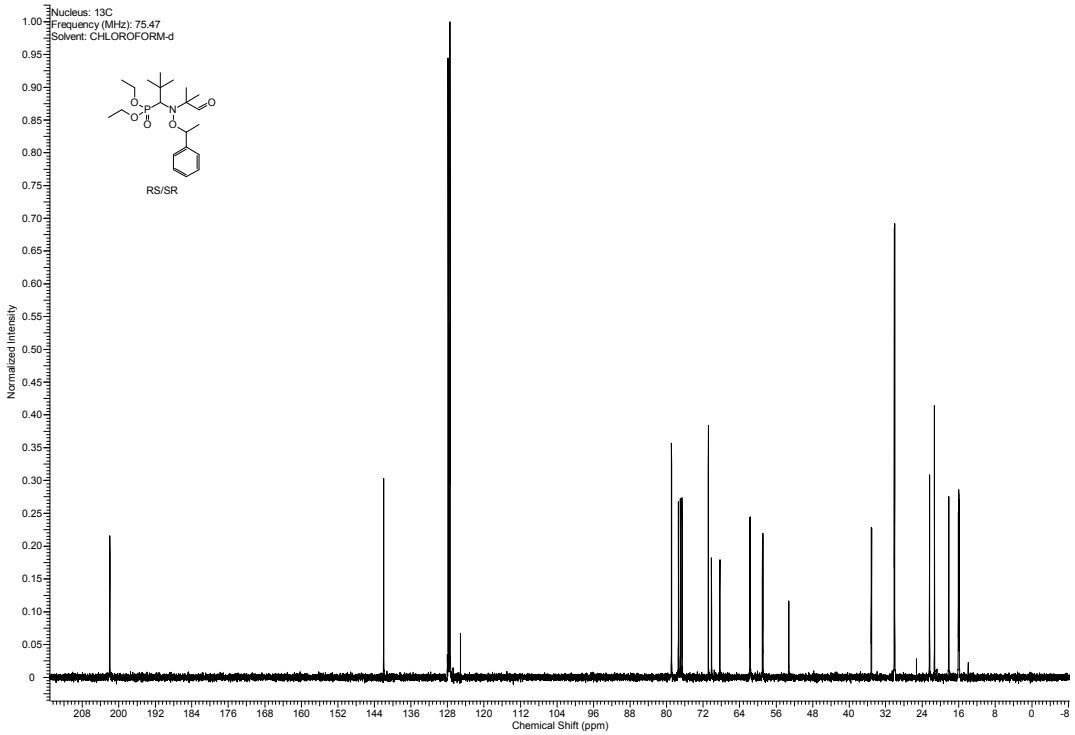


***RS/SR*-3:**

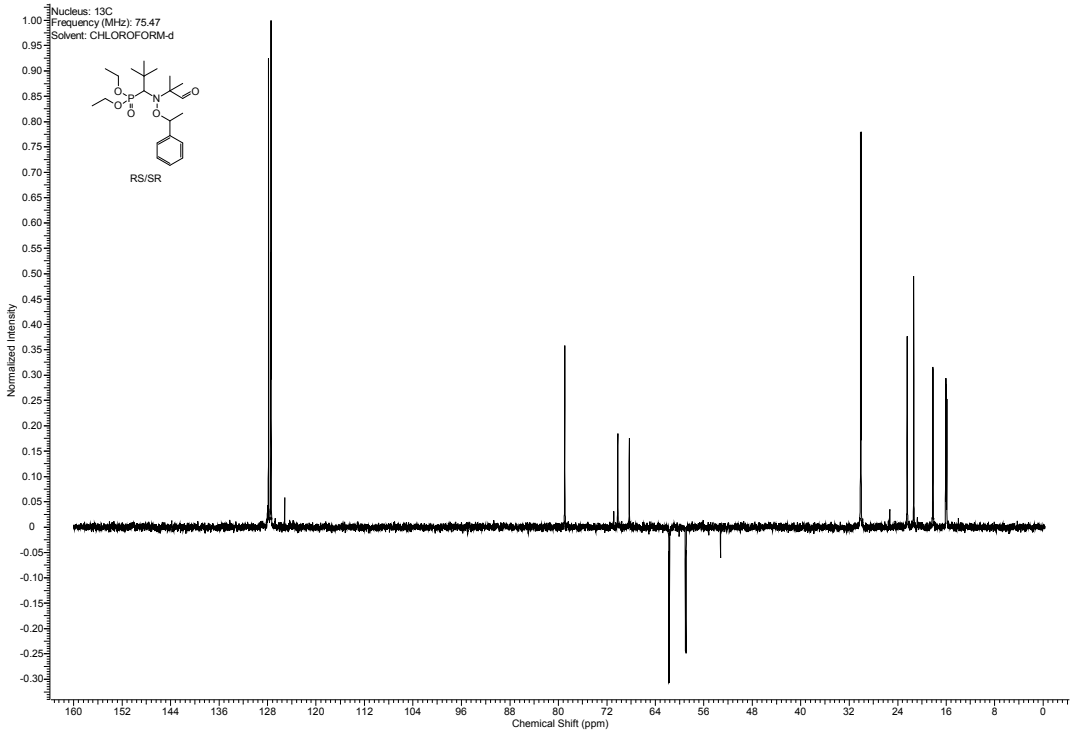
^{1}H-NMR (400 MHz, $CDCl_3$):



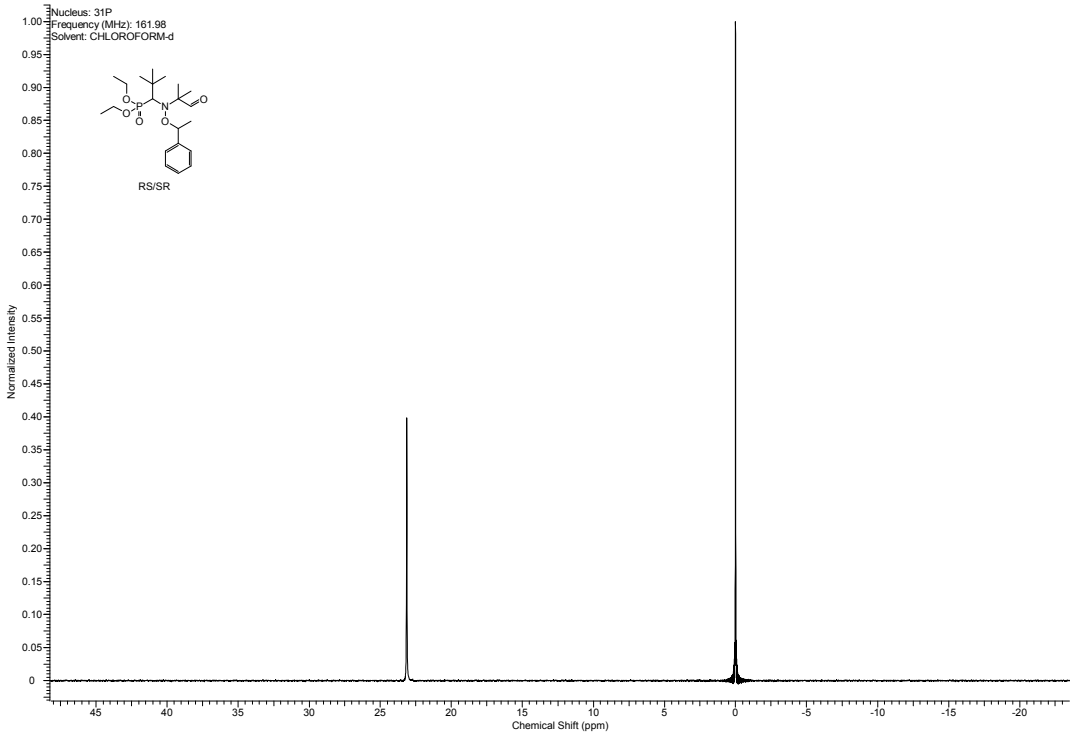
^{13}C{^{1}H}-NMR (75 MHz, $CDCl_3$):



DEPT135 (75 MHz, $CDCl_3$):

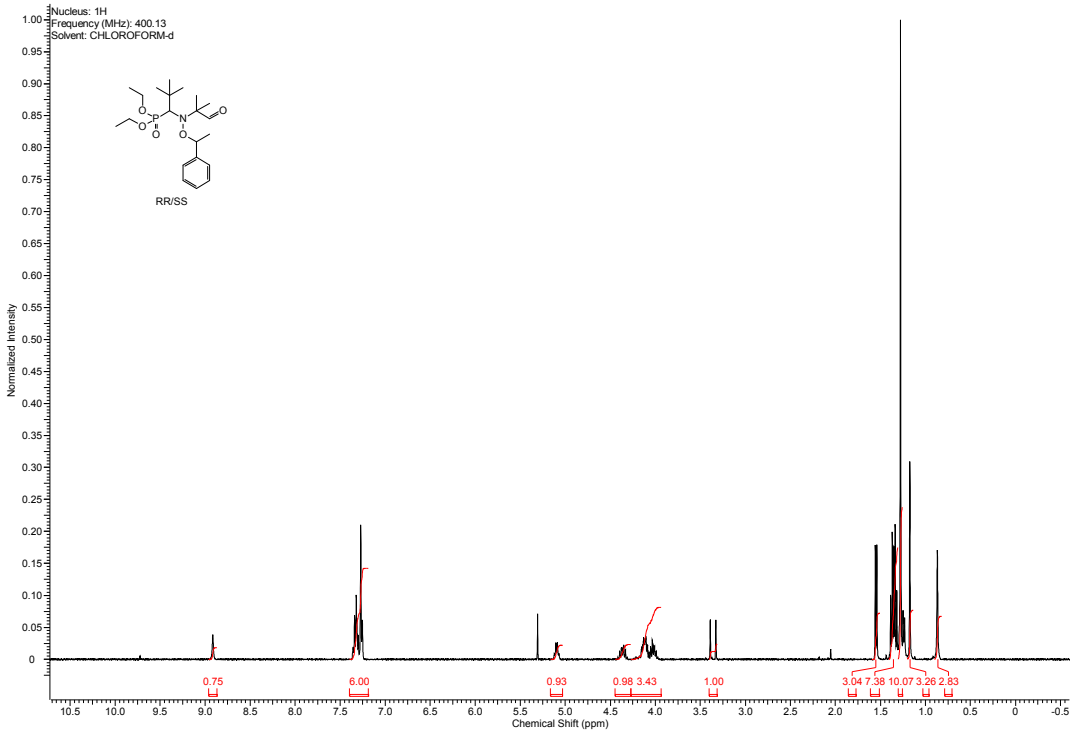


$^{31}P\{^{1}H\}$-NMR (162 MHz, $CDCl_3$):

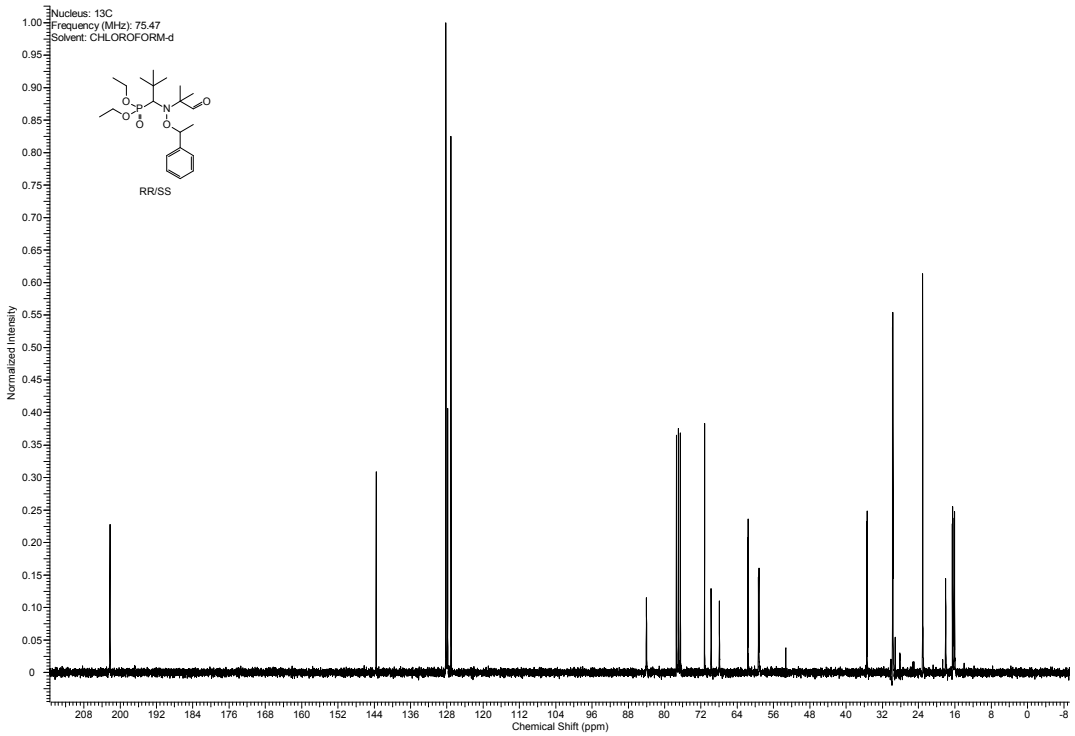


***RR/SS*-3:**

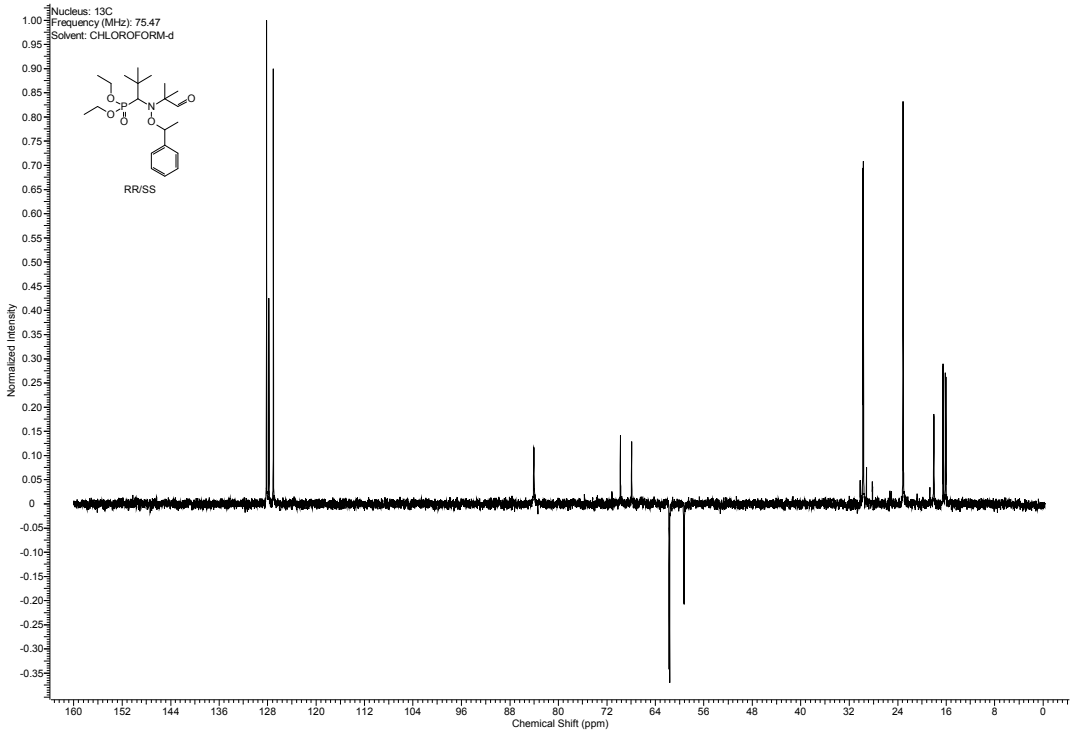
^{1}H-NMR (400 MHz, $CDCl_3$):



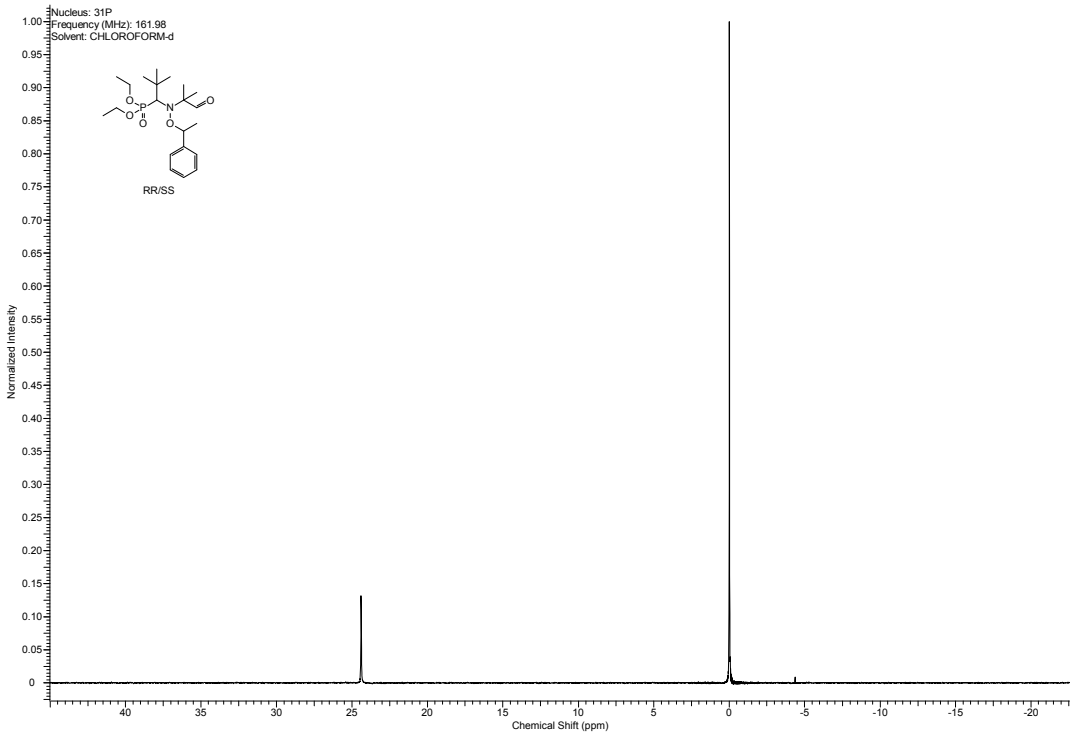
^{13}C{^{1}H}-NMR (75 MHz, $CDCl_3$):



DEPT135 (75 MHz, $CDCl_3$):

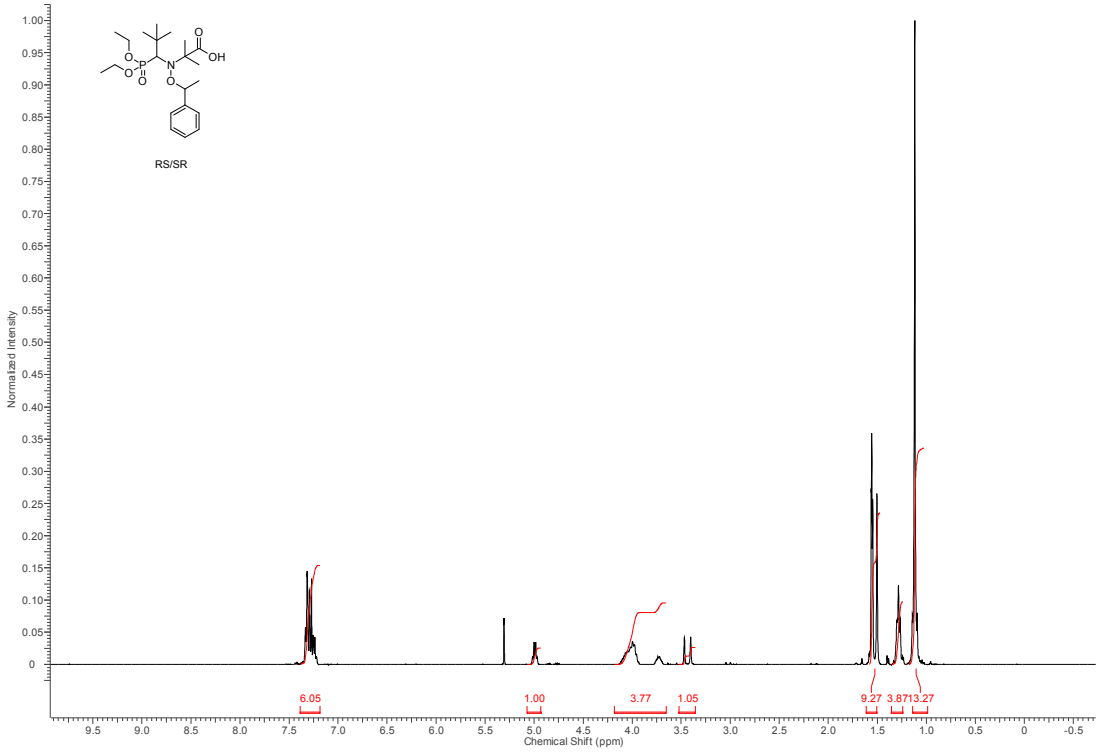


$^{31}P\{^{1}H\}$-NMR (162 MHz, $CDCl_3$):

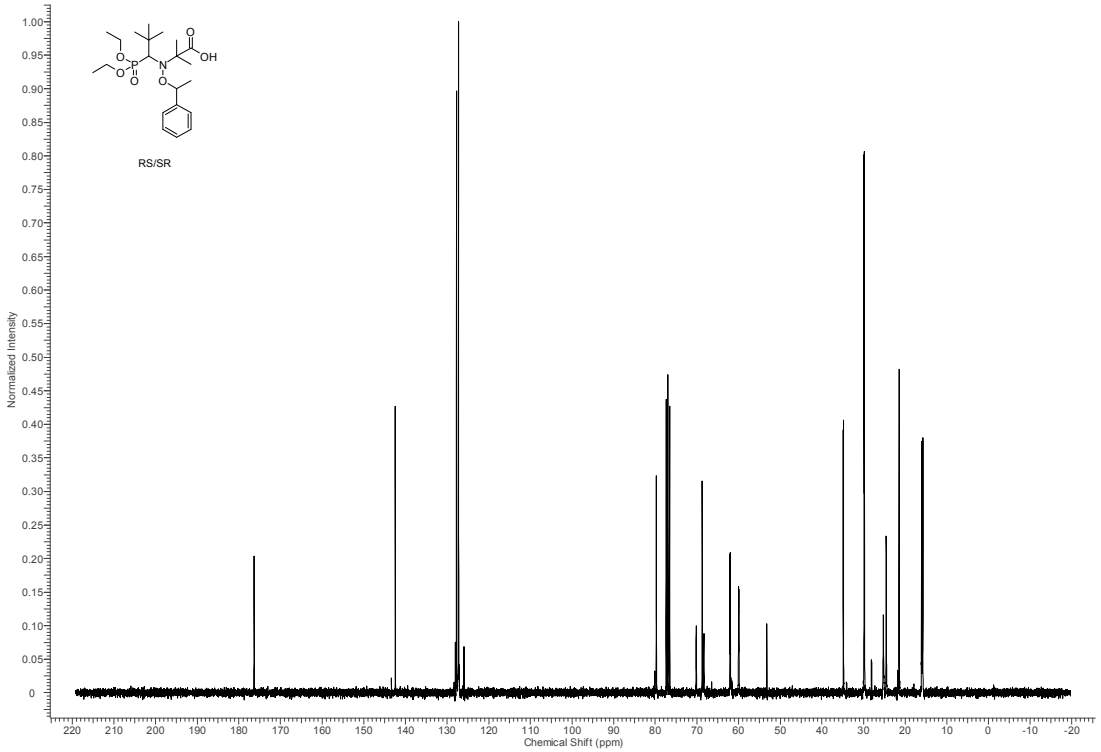


***RS/SR*-4:**

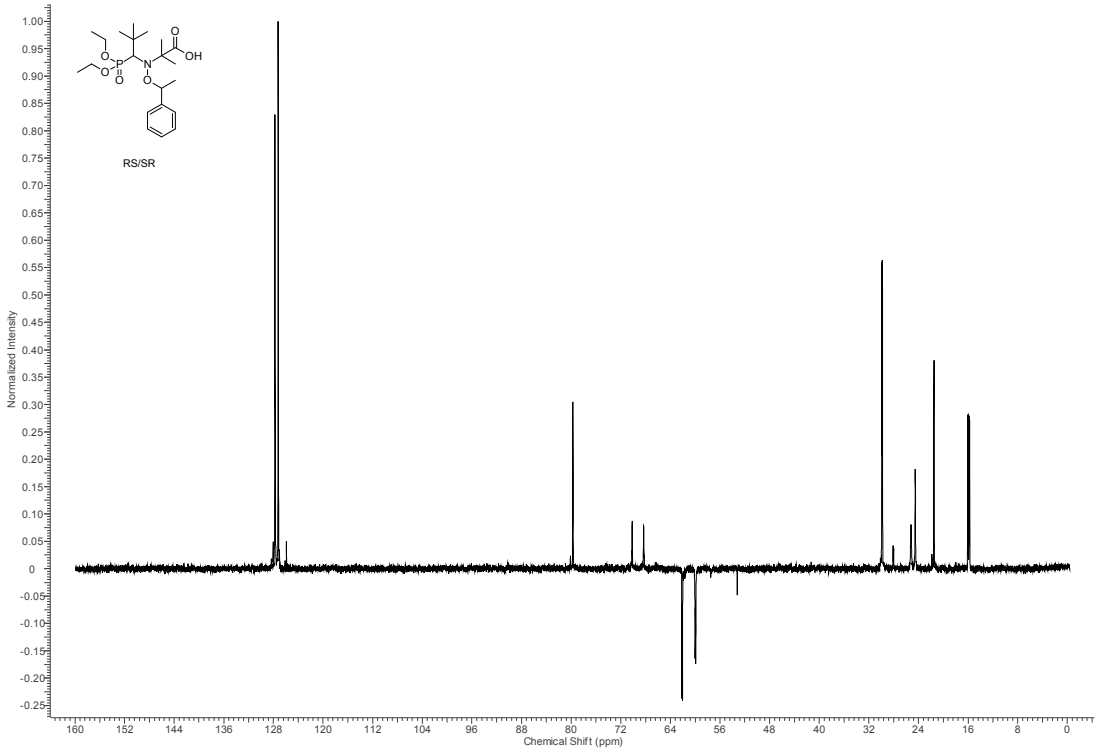
^{1}H-NMR (400 MHz, $CDCl_3$):



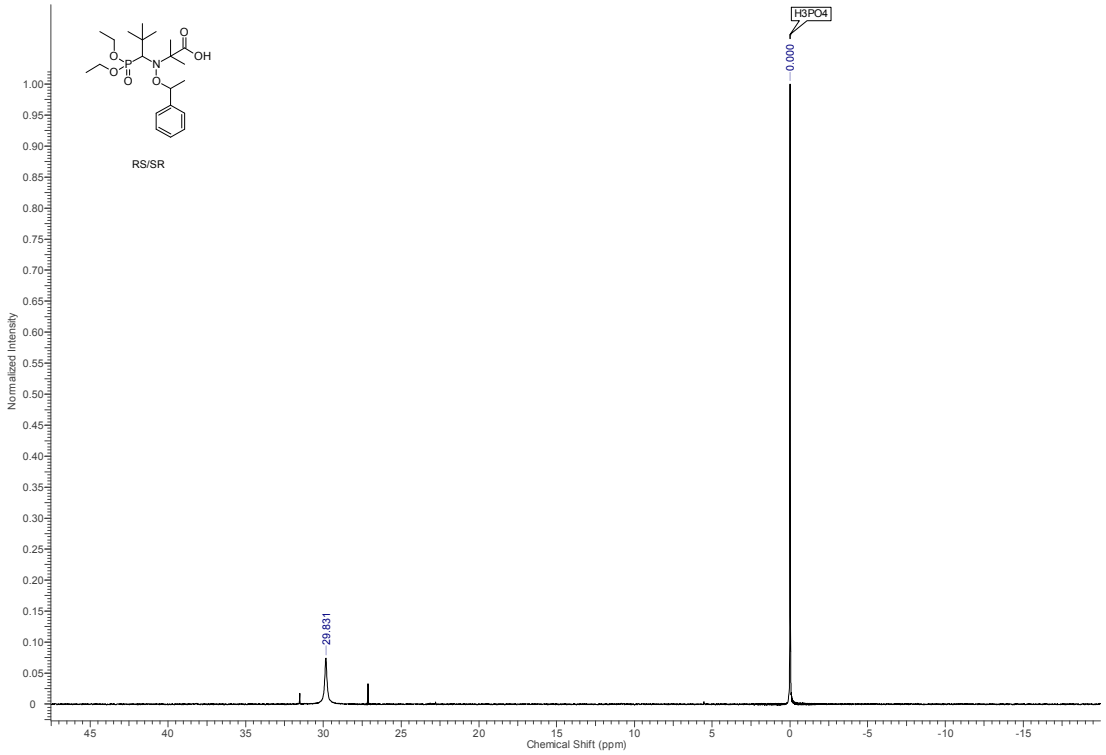
^{13}C{^{1}H}-NMR (75 MHz, $CDCl_3$):



DEPT135 (75 MHz, $CDCl_3$):

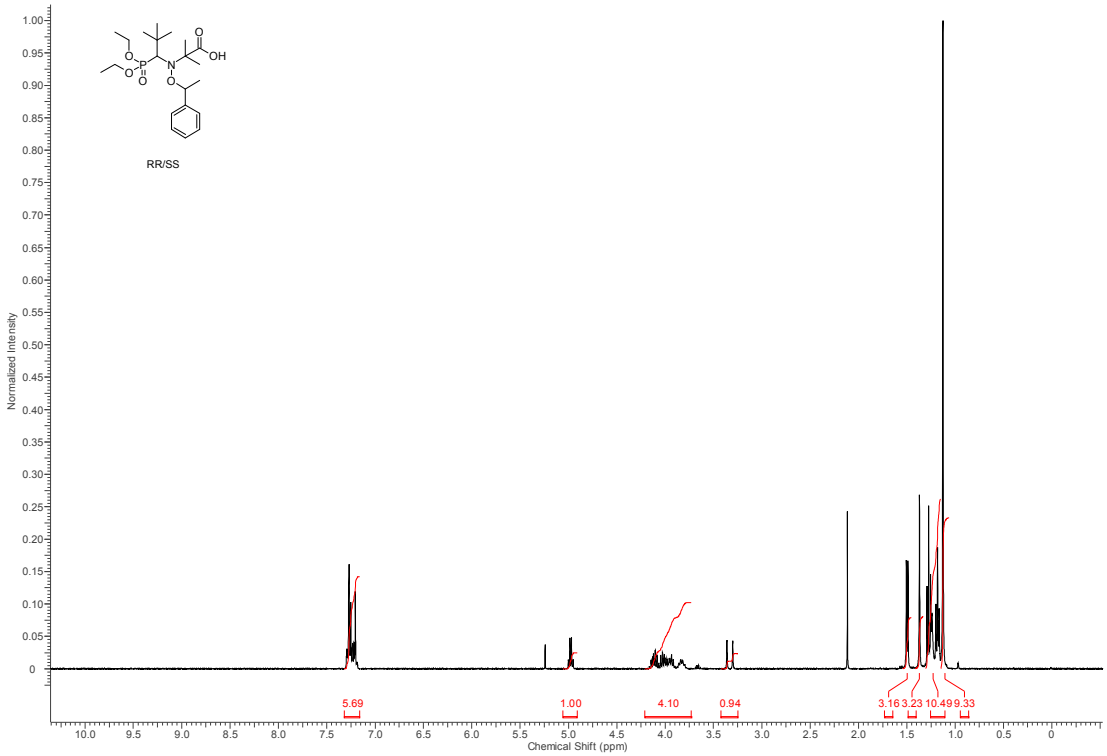


$^{31}P\{^{1}H\}$-NMR (162 MHz, $CDCl_3$):

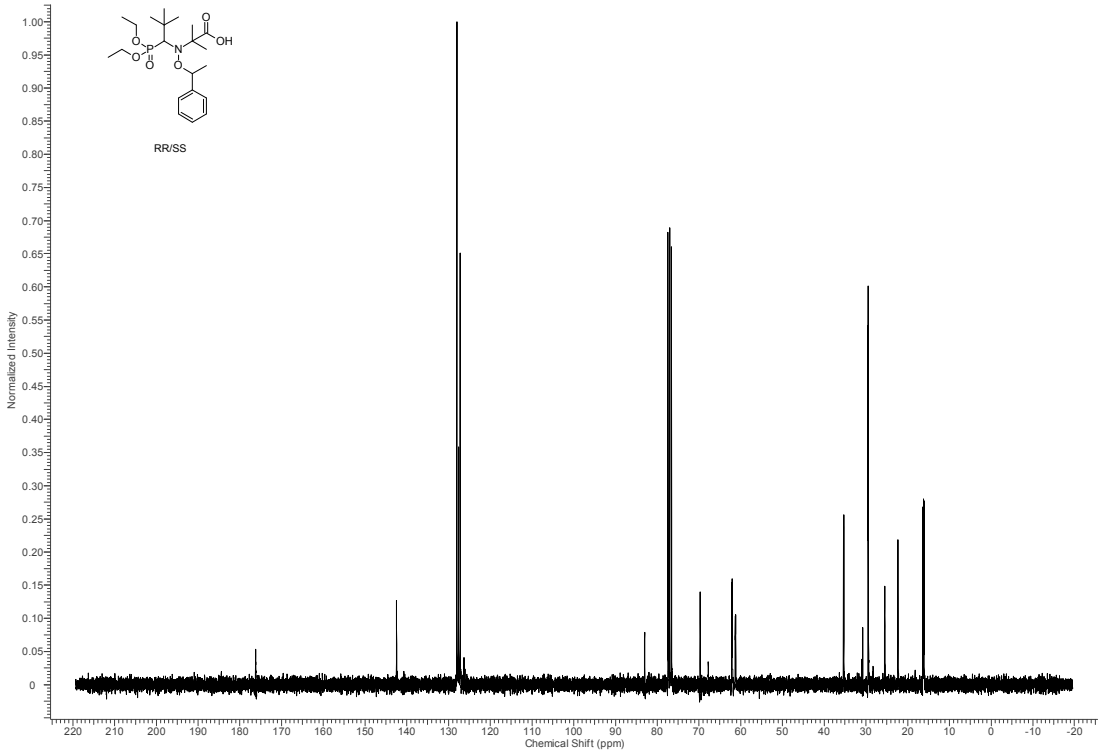


***RR/SS*-4:**

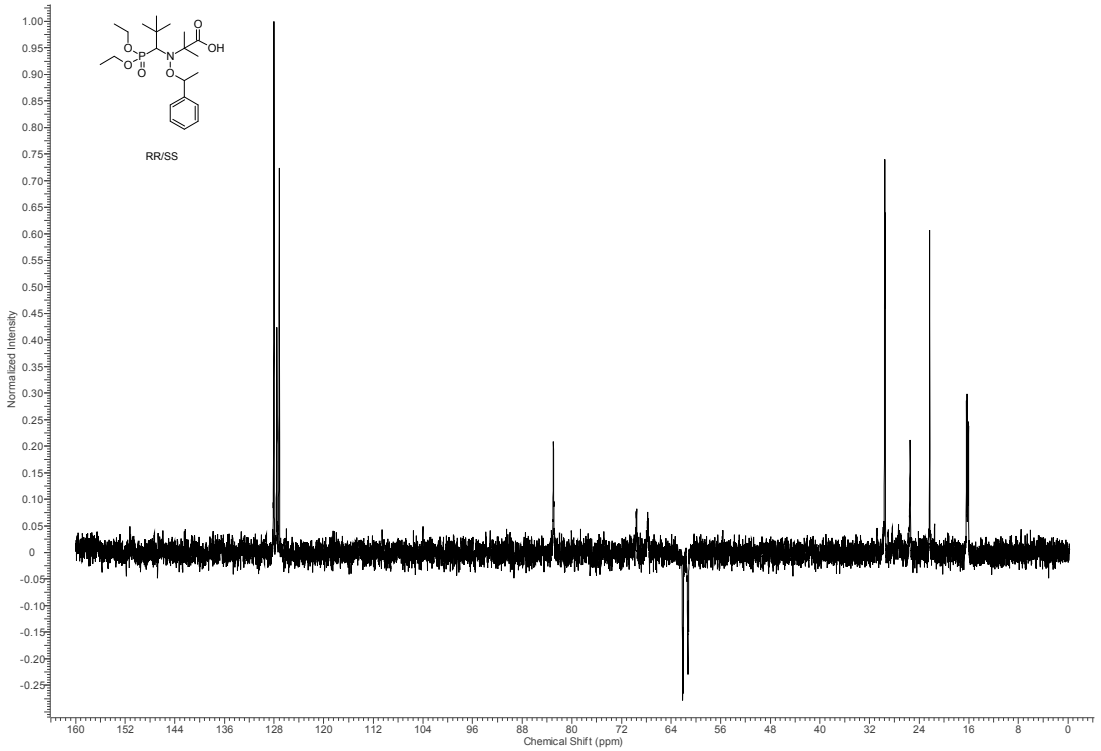
^{1}H-NMR (400 MHz, $CDCl_3$):



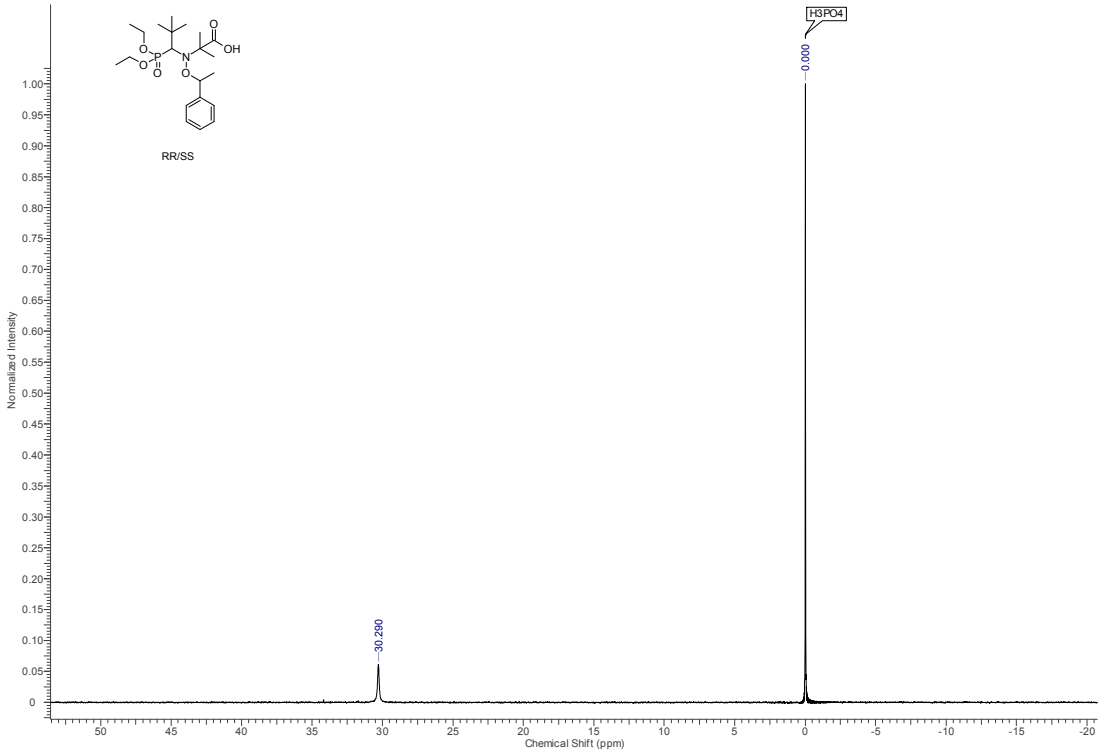
^{13}C{^{1}H}-NMR (75 MHz, $CDCl_3$):



DEPT135 (75 MHz, $CDCl_3$):

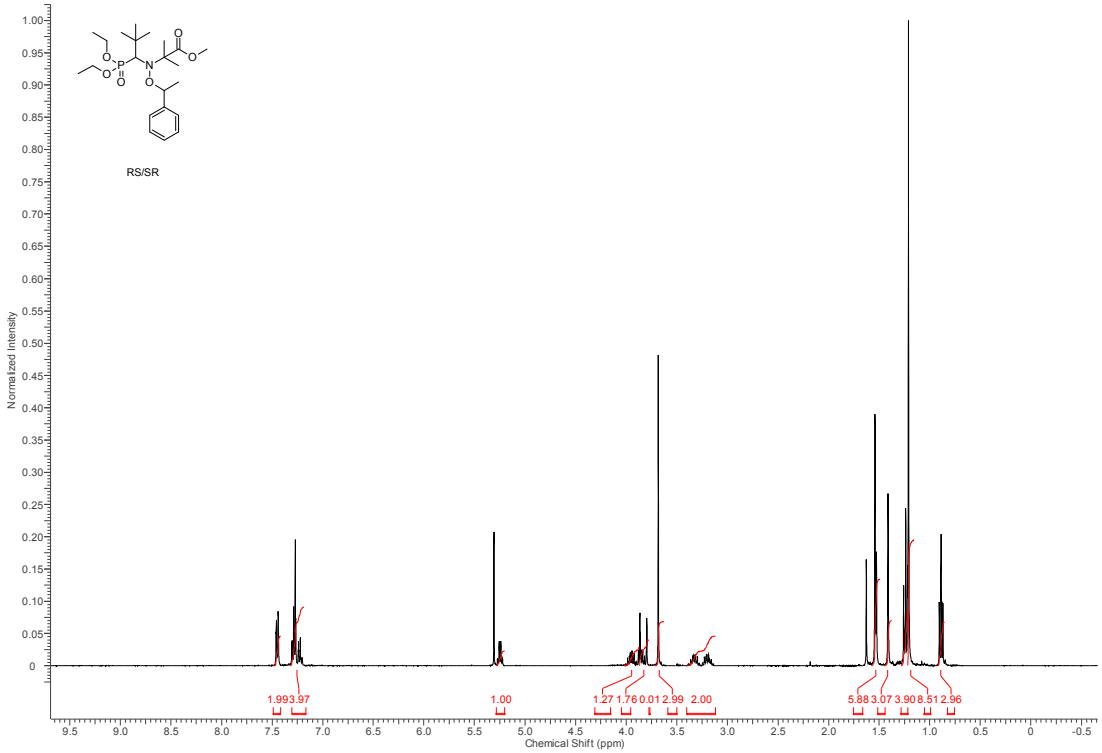


$^{31}P\{^{1}H\}$-NMR (162 MHz, $CDCl_3$):

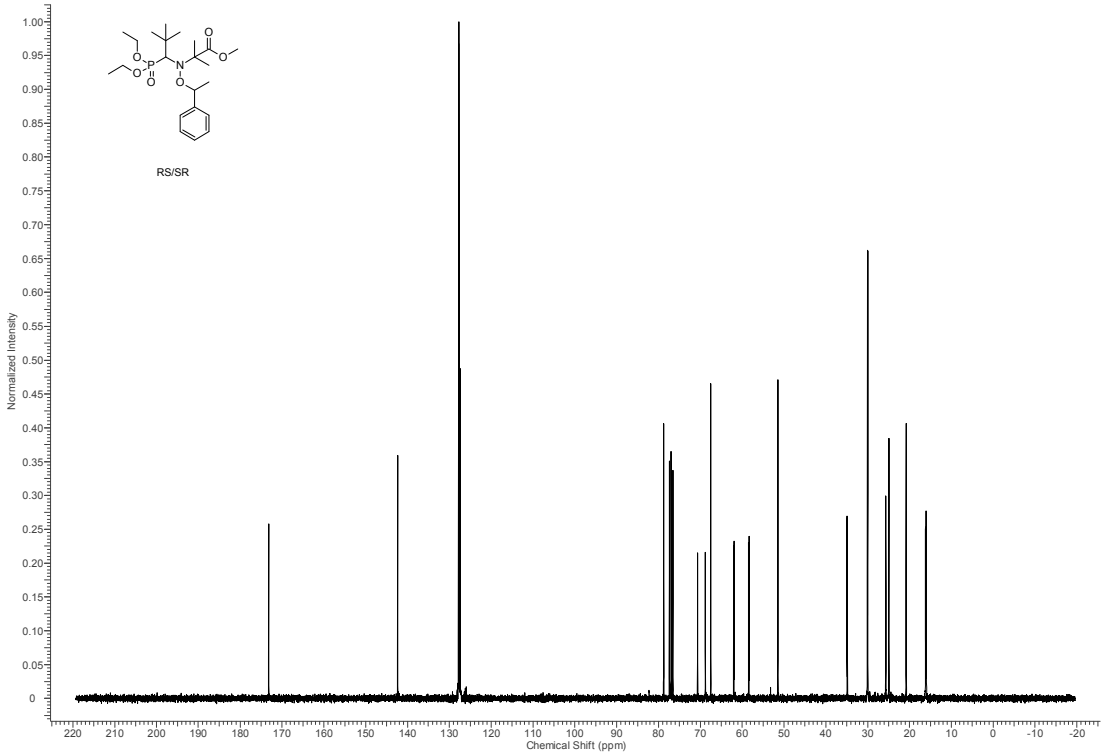


***RS/SR*-5:**

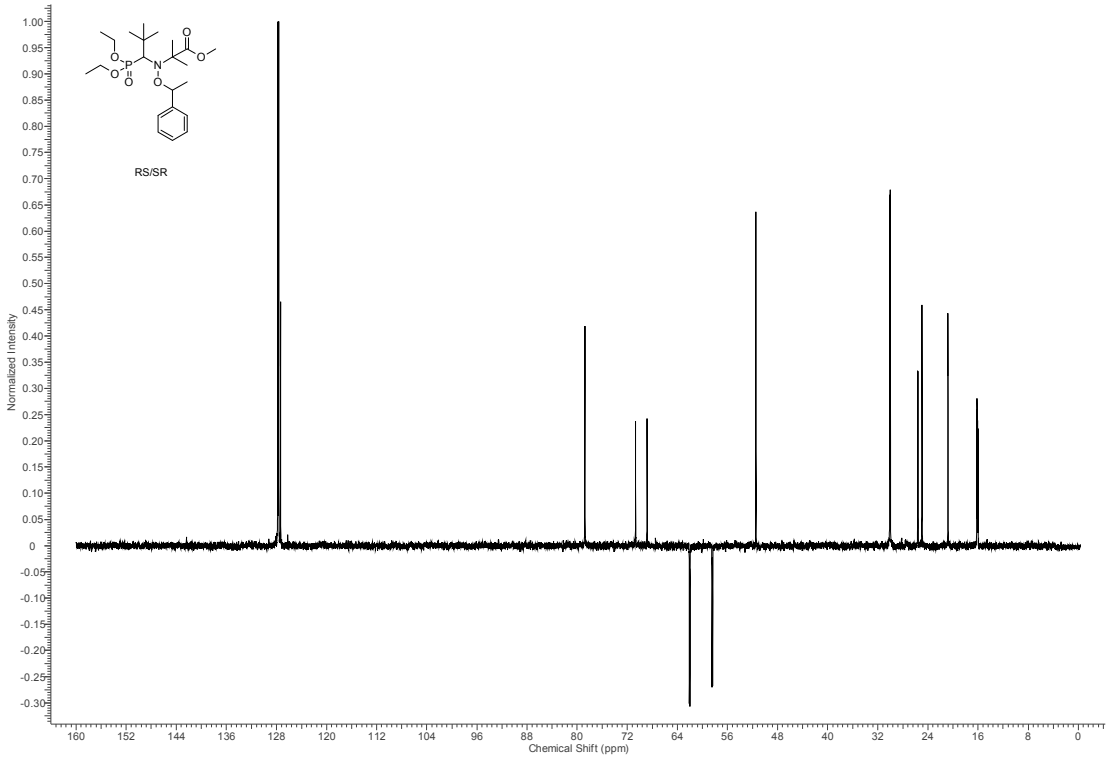
^{1}H-NMR (400 MHz, $CDCl_3$):



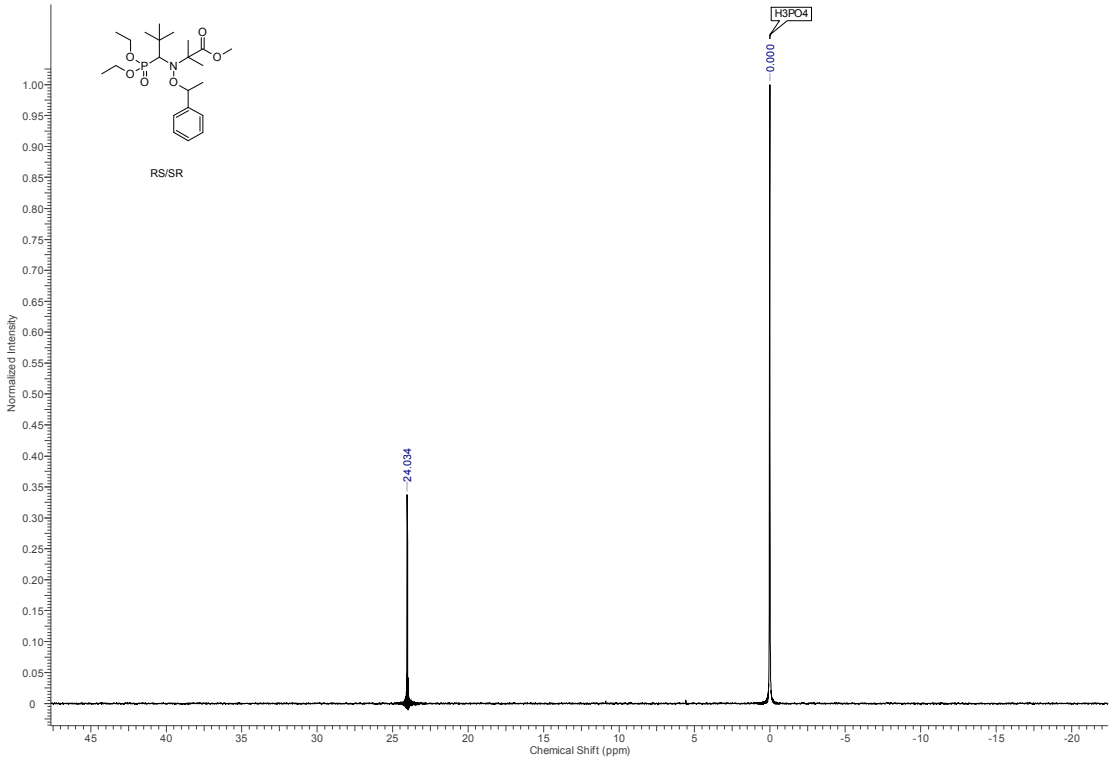
^{13}C{^{1}H}-NMR (75 MHz, $CDCl_3$):



DEPT135 (75 MHz, $CDCl_3$):

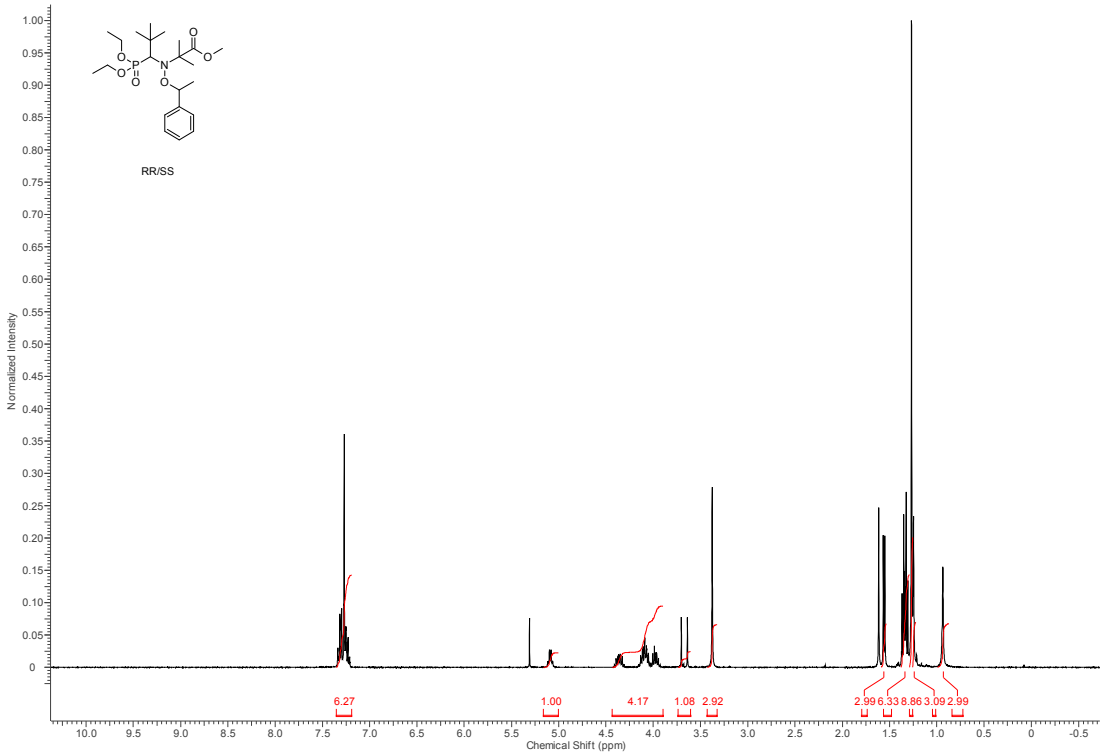


$^{31}P\{^{1}H\}$-NMR (162 MHz, $CDCl_3$):

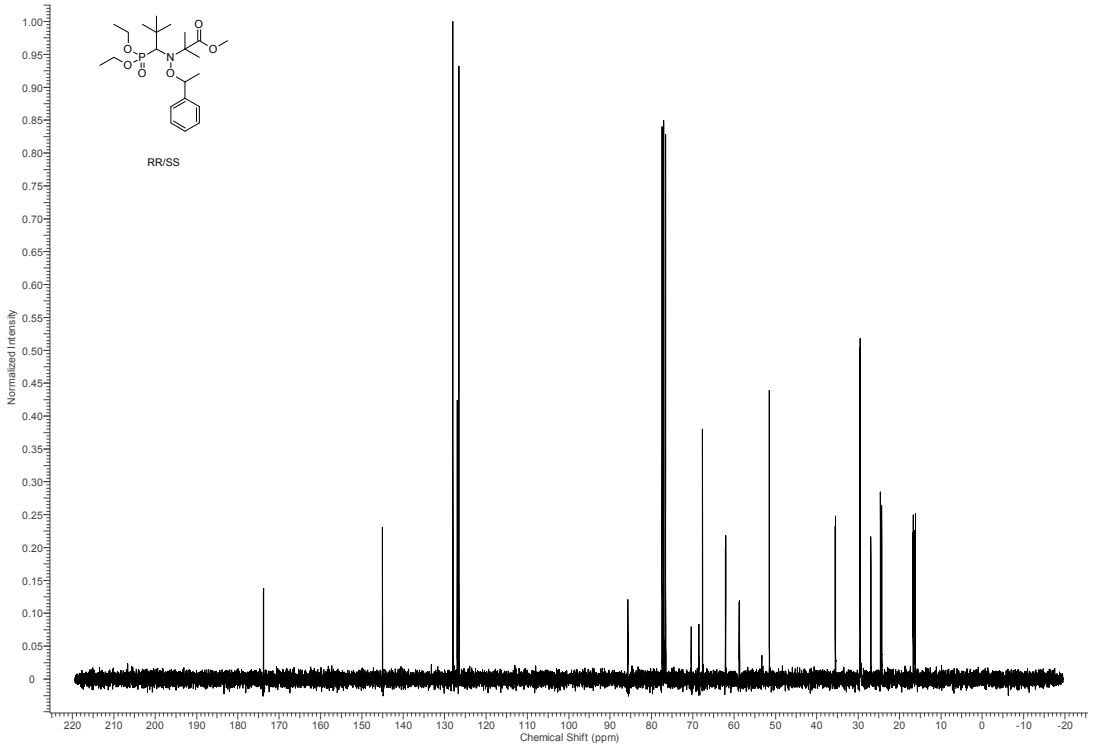


***RR/SS*-5:**

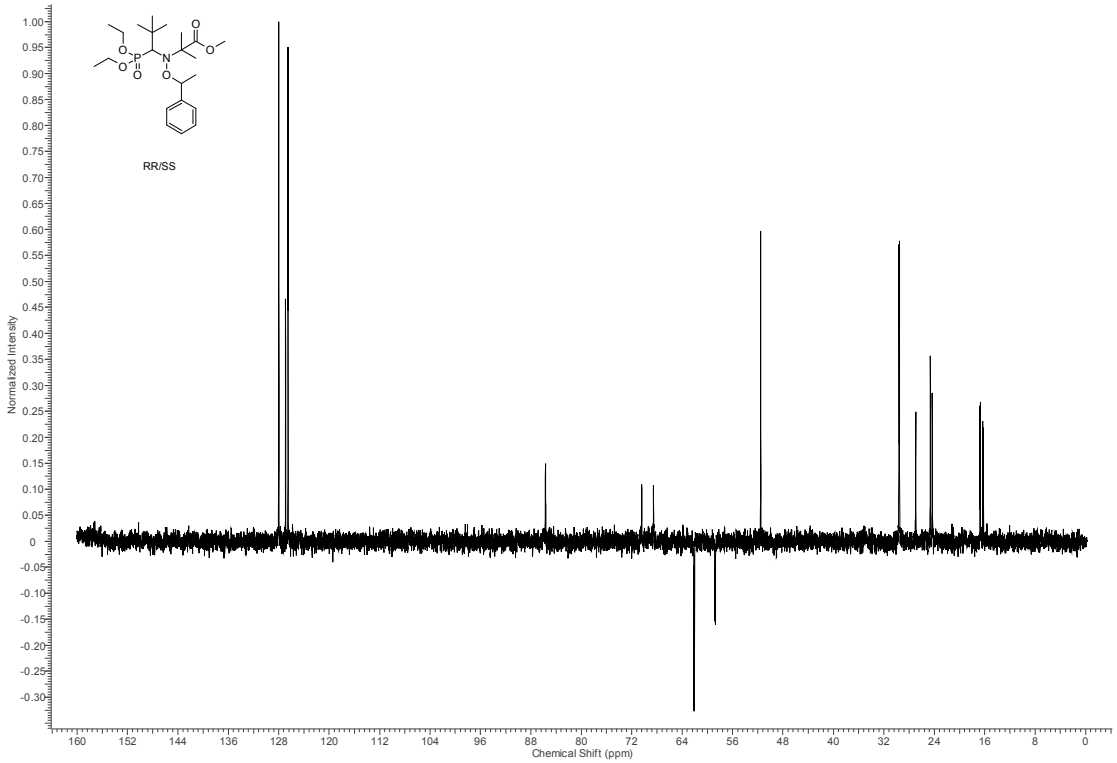
^{1}H-NMR (400 MHz, $CDCl_3$):



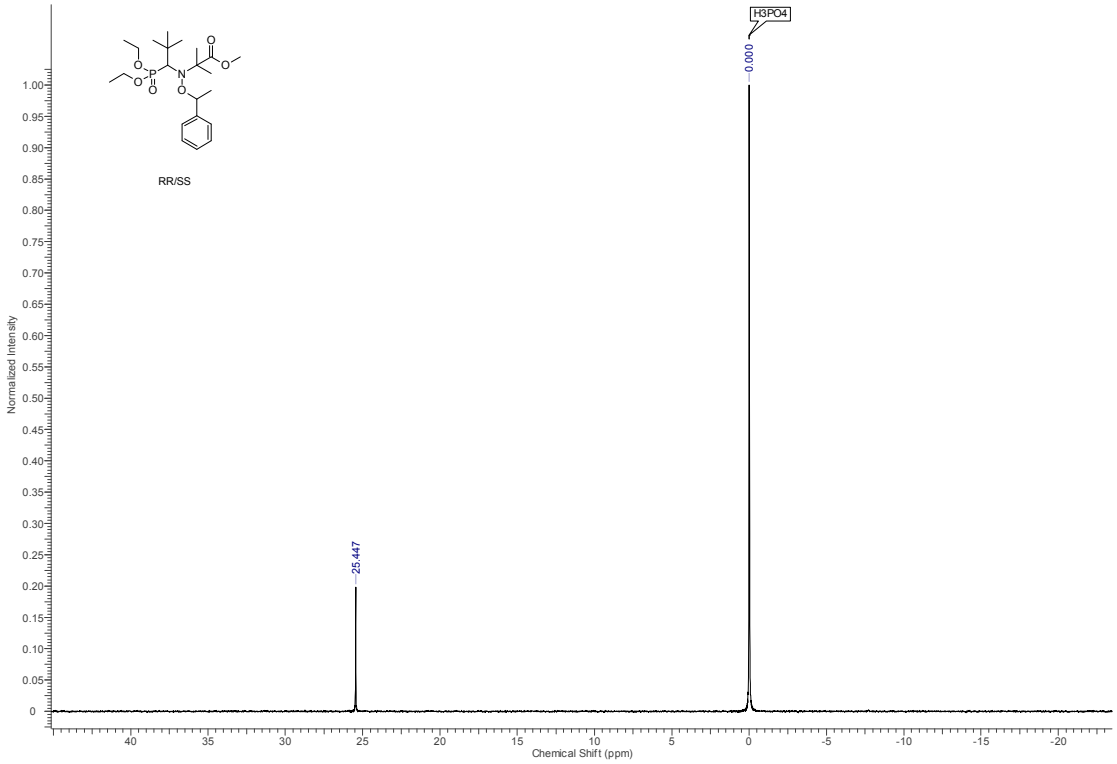
^{13}C{^{1}H}-NMR (75 MHz, $CDCl_3$):



DEPT135 (75 MHz, $CDCl_3$):



$^{31}P\{^{1}H\}$-NMR (162 MHz, $CDCl_3$):



1. Kinetic Measurements

Values of the homolysis rate constant k_d of **2, 6** and **7** were determined by monitoring the concentration of nitroxide by EPR. A sample tube containing a solution of 10^{-4} M of each diastereoisomer **2** in *tert*-butylbenzene or in H_2O/MeOH (1:1) was set in EPR cavity and EPR spectra were recorded sequentially. The temperature in the cavity was controlled by temperature control unit. The k_d values of **3-5** were determined by monitoring the concentration of alkoxyamines by $^{31}P\{^1H\}$-NMR in the presence of TEMPO (2,2,6,6-tetramethylpiperidin-*N*-oxyl) as alkyl radicals scavenger during the heating of the corresponding alkoxyamines. A stock solution of 0.02 M of alkoxyamine in *tert*-butylbenzene or in H_2O/MeOH with 2 equiv. of TEMPO was prepared and sampled into 20 NMR tubes (0.5 ml in each probe). Buffer solutions (for pH = 1.4, 100 mM of KCl/HCl buffer solution, and for pH = 8.4 and 10.4; 100 mM of Na2CO3/NaHCO3 buffer solution) were used for specific pH condition instead of H_2O. They were sunk in a pre-heated oil bath, withdrawn at various time intervals and quenched in icy-water bath. Then, 0.1 ml of C_6D_6 with $(EtO)_3PO$ (0.002 M) as internal standard (δ = 0 ppm) was added to each tubes. $^{31}P\{^1H\}$-NMR signal was recorded with conventional conditions on a 400 MHz NMR spectrometer.

In general, more than 90% conversion was reached. The value of activation energy was evaluated with the average frequency factor $A = 2.4 \times 10^{14}$ s^{-1}.

2. Intramolecular H-bonding (IHB)

The effect of IHB was measured with mixed solution of same relative configuration of **2** and **7**, or **4** and **5**. $^{31}P\{^1H\}$-NMR spectra were recorded on 400 MHz spectrometer. A capillary of 85% H_3PO_4 was used as an internal standard (δ = 0 ppm).

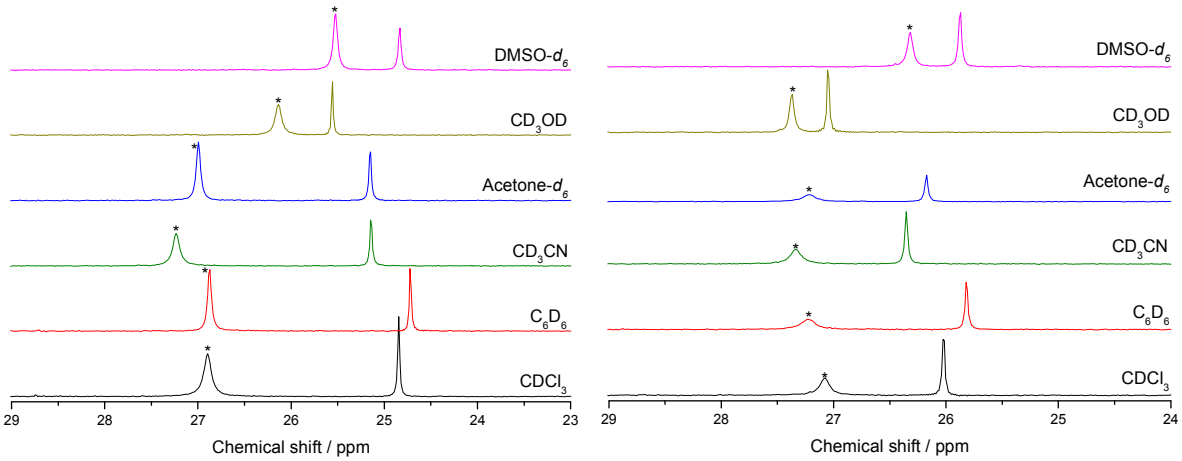


Figure 1SI. IHB effect of **2** and **7**, left: RS/SR, right: RR/SS. The peaks of **2** are marked with star.

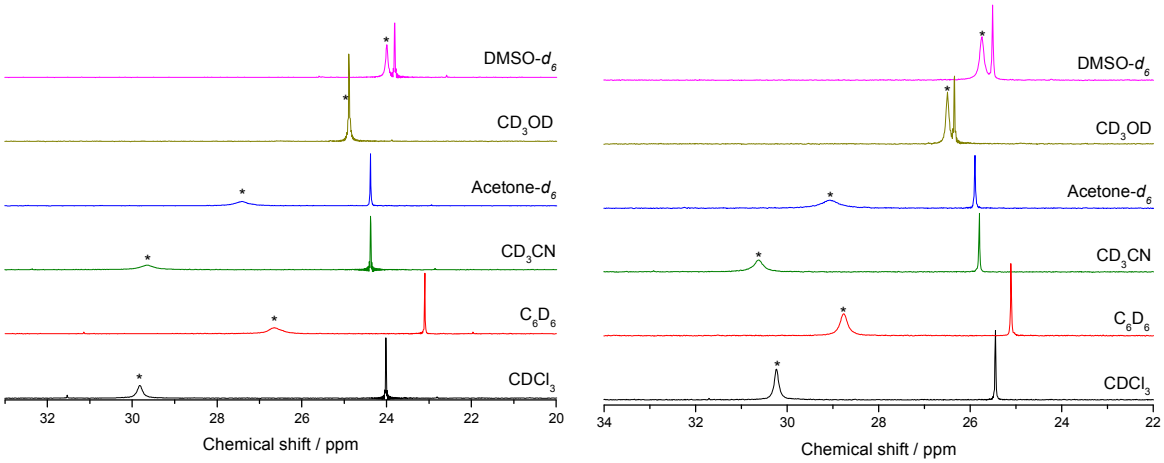


Figure 2SI. IHB effect of **4** and **5**, left: RS/SR, right: RR/SS. The peaks of **4** are marked with star.

3. pKa measurement

pKa value of **4** was measured by monitoring the dependency of ^{1}H NMR chemical shift in various pH*. A solution of 0.01 M **4** in D_2O/CD_3OD (1/1) was used. The pH* values were adjusted with DCl and NaOD and converted to pH values with the equation pH = 0.929 pH* + 0.42[1]. ^{1}H-NMR spectra were recorded on 400 MHz spectrometer.

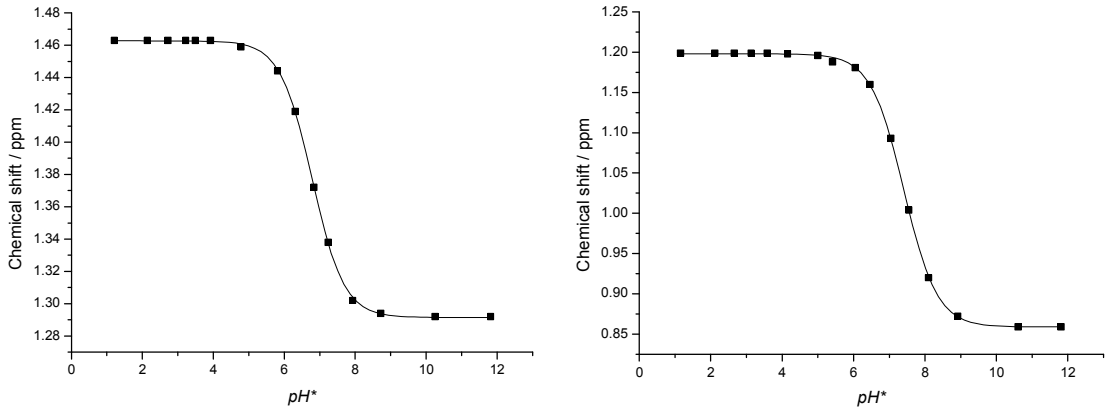
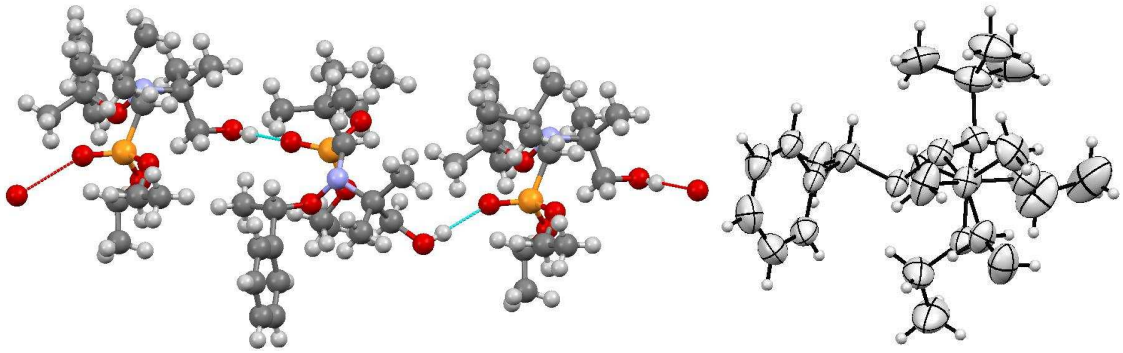


Figure 3SI. pKa measurement of **4**, left: RS/SR, right: RR/SS.

4. X-ray Structures

The ellipsoid contour percent is given at 50% for all ORTEP plots below.

(*RR/SS*)-2



Dolomanov, O.V., Bourhis, L.J., Gildea, R.J, Howard, J.A.K. & Puschmann, H.

(2009), J. Appl. Cryst. 42, 339-341.

Sheldrick, G.M. (2008). Acta Cryst. A64, 112-122.

_chemical_name_common ?

_chemical_name_systematic ?

_chemical_formula_moiety 'C21 H38 N O5 P'

_chemical_formula_sum 'C21 H38 N O5 P'

_chemical_formula_weight 415.49

_chemical_melting_point ?

loop_

_atom_type_symbol

_atom_type_description

_atom_type_scat_dispersion_real

_atom_type_scat_dispersion_imag

_atom_type_scat_source

'C' 'C' 0.0033 0.0016 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

'H' 'H' 0.0000 0.0000 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

'N' 'N' 0.0061 0.0033 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

'O' 'O' 0.0106 0.0060 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

'P' 'P' 0.1023 0.0942 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

_shelx_space_group_comment

The symmetry employed for this shelxl refinement is uniquely defined

by the following loop, which should always be used as a source of

symmetry information in preference to the above space-group names.

They are only intended as comments.

_space_group_crystal_system 'monoclinic'

_space_group_IT_number 14

_space_group_name_H-M_alt 'P 1 21/c 1'

_space_group_name_Hall '-P 2ybc'

loop_

_space_group_symop_operation_xyz

'x, y, z'

'-x, y+1/2, -z+1/2'

'-x, -y, -z'

'x, -y-1/2, z-1/2'

_cell_length_a 14.2019(10)

_cell_length_b 12.1086(7)

_cell_length_c 14.2955(8)

_cell_angle_alpha 90

_cell_angle_beta 102.371(6)

_cell_angle_gamma 90

_cell_volume 2401.3(3)

_cell_formula_units_Z 4

_cell_measurement_reflns_used 4143

_cell_measurement_temperature 293(2)

_cell_measurement_theta_max 23.0320

_cell_measurement_theta_min 3.9320

_shelx_estimated_absorpt_T_max ?

_shelx_estimated_absorpt_T_min ?

_exptl_absorpt_coefficient_mu 0.143

_exptl_absorpt_correction_T_max 1.00000

_exptl_absorpt_correction_T_min 0.90108

_exptl_absorpt_correction_type multi-scan

_exptl_absorpt_process_details

CrysAlisPro, Agilent Technologies,

Version 1.171.37.35 (release 13-08-2014 CrysAlis171 .NET)

(compiled Aug 13 2014,18:06:01)

Empirical absorption correction using spherical harmonics,

implemented in SCALE3 ABSPACK scaling algorithm.

_exptl_absorpt_special_details ?

_exptl_crystal_colour colourless

_exptl_crystal_colour_primary colourless

_exptl_crystal_density_diffrn 1.149

_exptl_crystal_density_meas ?

_exptl_crystal_density_method ?

_exptl_crystal_description "

_exptl_crystal_F_000 904

_exptl_crystal_size_max 0.22

_exptl_crystal_size_mid 0.2

_exptl_crystal_size_min 0.14

_exptl_transmission_factor_max ?

_exptl_transmission_factor_min ?

_diffrn_reflns_av_R_equivalents 0.0465

_diffrn_reflns_av_unetI/netI 0.0507

_diffrn_reflns_Laue_measured_fraction_full 0.998

_diffrn_reflns_Laue_measured_fraction_max 0.855

_diffrn_reflns_limit_h_max 17

_diffrn_reflns_limit_h_min -18

_diffrn_reflns_limit_k_max 16

_diffrn_reflns_limit_k_min -14

_diffrn_reflns_limit_l_max 19

_diffrn_reflns_limit_l_min -19

_diffrn_reflns_number 20231

_diffrn_reflns_point_group_measured_fraction_full 0.998

_diffrn_reflns_point_group_measured_fraction_max 0.855

_diffrn_reflns_theta_full 25.242

_diffrn_reflns_theta_max 28.897

_diffrn_reflns_theta_min 3.368

_diffrn_ambient_temperature 293(2)

```
_diffrn_detector                        'CCD plate'
_diffrn_detector_area_resol_mean    5.3048
_diffrn_detector_type                   AtlasS2
_diffrn_measured_fraction_theta_full 0.998
_diffrn_measured_fraction_theta_max 0.855
_diffrn_measurement_details
#__ type_ start__ end____ width___ exp.time_
   1 omega    -16.00     86.00     1.0000     10.0000
omega____ theta____ kappa____ phi______ frames
      -           10.6548     38.0000     30.0000 102
#__ type_ start__ end____ width___ exp.time_
   2 omega    -10.00     16.00     1.0000     10.0000
omega____ theta____ kappa____ phi______ frames
      -           -9.5611     57.0000    -30.0000 26
#__ type_ start__ end____ width___ exp.time_
   3 omega     -7.00     32.00     1.0000     10.0000
omega____ theta____ kappa____ phi______ frames
      -           -9.5611    -99.0000 -150.0000 39
#__ type_ start__ end____ width___ exp.time_
```

```
    4 omega    -18.00      52.00      1.0000      10.0000
omega____ theta____ kappa____ phi______ frames
        -            10.6548    -99.0000 -150.0000 70
#__ type_ start__ end____ width___ exp.time_
    5 omega    -68.00      44.00      1.0000      10.0000
omega____ theta____ kappa____ phi______ frames
        -            10.6548    -77.0000      90.0000 112
_diffrn_measurement_device              'four-circle diffractometer'
_diffrn_measurement_device_type      'SuperNova, Dual, Cu at zero, AtlasS2'
_diffrn_measurement_method              '¥w scans'
_diffrn_orient_matrix_UB_11             0.0358643000
_diffrn_orient_matrix_UB_12             -0.0401807000
_diffrn_orient_matrix_UB_13             -0.0021367000
_diffrn_orient_matrix_UB_21             -0.0364168000
_diffrn_orient_matrix_UB_22             -0.0396106000
_diffrn_orient_matrix_UB_23             -0.0170052000
_diffrn_orient_matrix_UB_31             -0.0002958000
_diffrn_orient_matrix_UB_32             0.0158652000
_diffrn_orient_matrix_UB_33             -0.0478124000
```

_diffrn_radiation_monochromator mirror

_diffrn_radiation_probe x-ray

_diffrn_radiation_type MoK¥a

_diffrn_radiation_wavelength 0.71073

_diffrn_source 'SuperNova (Mo) X-ray Source'

_reflns_Friedel_coverage 0.000

_reflns_Friedel_fraction_full .

_reflns_Friedel_fraction_max .

_reflns_number_gt 3390

_reflns_number_total 5416

_reflns_odcompleteness_completeness 99.68

_reflns_odcompleteness_iscentric 1

_reflns_odcompleteness_theta 25.30

_reflns_special_details

Reflections were merged by SHELXL according to the crystal

class for the calculation of statistics and refinement.

_reflns_Friedel_fraction is defined as the number of unique

Friedel pairs measured divided by the number that would be

possible theoretically, ignoring centric projections and

systematic absences.

_reflns_threshold_expression 'I > 2¥s(I)'

_computing_cell_refinement

CrysAlisPro, Agilent Technologies,

Version 1.171.37.35 (release 13-08-2014 CrysAlis171 .NET)

(compiled Aug 13 2014,18:06:01)

_computing_data_collection

CrysAlisPro, Agilent Technologies,

Version 1.171.37.35 (release 13-08-2014 CrysAlis171 .NET)

(compiled Aug 13 2014,18:06:01)

_computing_data_reduction

CrysAlisPro, Agilent Technologies,

Version 1.171.37.35 (release 13-08-2014 CrysAlis171 .NET)

(compiled Aug 13 2014,18:06:01)

_computing_molecular_graphics 'Olex2 (Dolomanov et al., 2009)'

_computing_publication_material 'Olex2 (Dolomanov et al., 2009)'

_computing_structure_refinement 'ShelXL (Sheldrick, 2008)'

_computing_structure_solution ?

_refine_diff_density_max 0.684

_refine_diff_density_min -0.341

_refine_diff_density_rms 0.048

_refine_ls_extinction_coef .

_refine_ls_extinction_method none

_refine_ls_goodness_of_fit_ref 1.043

_refine_ls_hydrogen_treatment constr

_refine_ls_matrix_type full

_refine_ls_number_parameters 262

_refine_ls_number_reflns 5416

_refine_ls_number_restraints 0

_refine_ls_R_factor_all 0.1148

_refine_ls_R_factor_gt 0.0712

_refine_ls_restrained_S_all 1.043

_refine_ls_shift/su_max 0.000

_refine_ls_shift/su_mean 0.000

_refine_ls_structure_factor_coef Fsqd

_refine_ls_weighting_details

'w=1/[¥s^2^(Fo^2^)+(0.0834P)^2^+1.3513P] where P=(Fo^2^+2Fc^2^)/3'

_refine_ls_weighting_scheme calc

_refine_ls_wR_factor_gt 0.1768

_refine_ls_wR_factor_ref 0.2049

_refine_special_details ?

_olex2_refinement_description

1. Fixed Uiso

At 1.2 times of:

All C(H) groups, All C(H,H) groups

At 1.5 times of:

All C(H,H,H) groups, All O(H) groups

2.a Ternary CH refined with riding coordinates:

C1(H1), C2(H2)

2.b Secondary CH2 refined with riding coordinates:

C11(H11A,H11B), C18(H18A,H18B), C20(H20A,H20B)

2.c Aromatic/amide H refined with riding coordinates:

C4(H4), C5(H5A), C6(H6), C7(H7), C8(H8)

2.d Idealised Me refined as rotating group:

C9(H9A,H9B,H9C), C12(H12A,H12B,H12C), C13(H13A,H13B,H13C), C15(H15A,H15B,

H15C), C16(H16A,H16B,H16C), C17(H17A,H17B,H17C), C19(H19A,H19B,H19C), C21(H21A,

H21B,H21C)

2.e Idealised tetrahedral OH refined as rotating group:

O5(H5)

;

_atom_sites_solution_hydrogens geom

_atom_sites_solution_primary ?

_atom_sites_solution_secondary ?

loop_

_atom_site_label

_atom_site_type_symbol

_atom_site_fract_x

_atom_site_fract_y

_atom_site_fract_z

_atom_site_U_iso_or_equiv

_atom_site_adp_type

_atom_site_occupancy

_atom_site_site_symmetry_order

_atom_site_calc_flag

_atom_site_refinement_flags_posn

_atom_site_refinement_flags_adp

_atom_site_refinement_flags_occupancy

_atom_site_disorder_assembly

_atom_site_disorder_group

P1 P 0.25758(6) 0.81369(6) 0.36420(5) 0.0494(3) Uani 1 1 d . . . . .

O1 O 0.26506(19) 0.84200(19) 0.26660(14) 0.0731(7) Uani 1 1 d . . . . .

O2 O 0.22016(17) 0.91106(16) 0.41985(15) 0.0626(6) Uani 1 1 d . . . . .

O3 O 0.35654(14) 0.78696(17) 0.43242(13) 0.0547(5) Uani 1 1 d . . . . .

O4 O 0.28539(13) 0.58992(15) 0.33874(12) 0.0451(5) Uani 1 1 d . . . . .

O5 O 0.3315(2) 0.5332(2) 0.63304(16) 0.0856(8) Uani 1 1 d . . . . .

H5 H 0.3147 0.5710 0.6742 0.128 Uiso 1 1 calc GR . . . .

N1 N 0.19942(16) 0.59283(18) 0.37911(15) 0.0441(6) Uani 1 1 d . . . . .

C1 C 0.1668(2) 0.7098(2) 0.3760(2) 0.0481(7) Uani 1 1 d . . . . .

H1 H 0.1516 0.7236 0.4386 0.058 Uiso 1 1 calc R . . . .

C2 C 0.2725(2) 0.5272(3) 0.25045(19) 0.0536(7) Uani 1 1 d . . . . .

H2 H 0.2034 0.5185 0.2236 0.064 Uiso 1 1 calc R . . . .

C3 C 0.3189(2) 0.4144(2) 0.26382(18) 0.0498(7) Uani 1 1 d . . . . .

C4 C 0.4142(2) 0.3995(3) 0.3098(2) 0.0656(9) Uani 1 1 d . . . . .

H4 H 0.4510 0.4601 0.3355 0.079 Uiso 1 1 calc R . . . .

C5 C 0.4551(3) 0.2958(3) 0.3179(3) 0.0774(11) Uani 1 1 d . . . . .

H5A H 0.5187 0.2868 0.3505 0.093 Uiso 1 1 calc R . . . .

C6 C 0.4027(3) 0.2056(3) 0.2781(3) 0.0774(11) Uani 1 1 d . . . . .

H6 H 0.4308 0.1359 0.2828 0.093 Uiso 1 1 calc R . . . .

C7 C 0.3090(3) 0.2194(3) 0.2316(2) 0.0690(10) Uani 1 1 d . . . . .

H7 H 0.2732 0.1588 0.2042 0.083 Uiso 1 1 calc R . . . .

C8 C 0.2672(2) 0.3221(3) 0.2250(2) 0.0556(8) Uani 1 1 d . . . . .

H8 H 0.2029 0.3298 0.1939 0.067 Uiso 1 1 calc R . . . .

C9 C 0.3170(4) 0.5954(3) 0.1817(2) 0.0881(13) Uani 1 1 d . . . . .

H9A H 0.2867 0.6667 0.1732 0.132 Uiso 1 1 calc GR . . . .

H9B H 0.3077 0.5584 0.1211 0.132 Uiso 1 1 calc GR . . . .

H9C H 0.3848 0.6043 0.2076 0.132 Uiso 1 1 calc GR . . . .

C10 C 0.2236(2) 0.5387(2) 0.47676(18) 0.0463(7) Uani 1 1 d . . . . .

C11 C 0.3094(2) 0.5898(3) 0.54379(19) 0.0599(8) Uani 1 1 d . . . . .

H11A H 0.3648 0.5874 0.5143 0.072 Uiso 1 1 calc R . . . .

H11B H 0.2956 0.6667 0.5547 0.072 Uiso 1 1 calc R . . . .

C12 C 0.2419(3) 0.4174(3) 0.4612(2) 0.0763(11) Uani 1 1 d . . . . .

H12A H 0.1896 0.3878 0.4139 0.114 Uiso 1 1 calc GR . . . .

H12B H 0.2467 0.3780 0.5203 0.114 Uiso 1 1 calc GR . . . .

H12C H 0.3010 0.4095 0.4395 0.114 Uiso 1 1 calc GR . . . .

C13 C 0.1364(3) 0.5452(3) 0.5227(3) 0.0813(11) Uani 1 1 d . . . . .

H13A H 0.1220 0.6212 0.5328 0.122 Uiso 1 1 calc GR . . . .

H13B H 0.1503 0.5073 0.5831 0.122 Uiso 1 1 calc GR . . . .

H13C H 0.0820 0.5113 0.4813 0.122 Uiso 1 1 calc GR . . . .

C14 C 0.0682(2) 0.7267(3) 0.3006(3) 0.0679(9) Uani 1 1 d . . . . .

C15 C 0.0756(3) 0.6961(4) 0.1980(3) 0.0951(14) Uani 1 1 d . . . . .

H15A H 0.1309 0.7318 0.1829 0.143 Uiso 1 1 calc GR . . . .

H15B H 0.0185 0.7199 0.1539 0.143 Uiso 1 1 calc GR . . . .

H15C H 0.0823 0.6175 0.1933 0.143 Uiso 1 1 calc GR . . . .

C16 C 0.0336(3) 0.8467(4) 0.3007(4) 0.1048(15) Uani 1 1 d . . . . .

H16A H 0.0324 0.8685 0.3649 0.157 Uiso 1 1 calc GR . . . .

H16B H -0.0300 0.8527 0.2612 0.157 Uiso 1 1 calc GR . . . .

H16C H 0.0769 0.8938 0.2759 0.157 Uiso 1 1 calc GR . . . .

C17 C -0.0086(3) 0.6538(4) 0.3287(3) 0.0937(13) Uani 1 1 d . . . . .

H17A H 0.0108 0.5779 0.3285 0.140 Uiso 1 1 calc GR . . . .

H17B H -0.0686 0.6638 0.2836 0.140 Uiso 1 1 calc GR . . . .

H17C H -0.0163 0.6738 0.3916 0.140 Uiso 1 1 calc GR . . . .

C18 C 0.2530(4) 1.0218(3) 0.4108(4) 0.1090(16) Uani 1 1 d . . . . .

H18A H 0.2476 1.0393 0.3437 0.131 Uiso 1 1 calc R . . . .

H18B H 0.3203 1.0279 0.4429 0.131 Uiso 1 1 calc R . . . .

C19 C 0.1965(5) 1.0991(4) 0.4525(4) 0.138(2) Uani 1 1 d . . . . .

H19A H 0.1309 1.0973 0.4173 0.206 Uiso 1 1 calc GR . . . .

H19B H 0.2221 1.1721 0.4499 0.206 Uiso 1 1 calc GR . . . .

H19C H 0.1989 1.0792 0.5179 0.206 Uiso 1 1 calc GR . . . .

C20 C 0.4437(3) 0.7616(3) 0.3995(3) 0.0770(10) Uani 1 1 d . . . . .

H20A H 0.4291 0.7569 0.3302 0.092 Uiso 1 1 calc R . . . .

H20B H 0.4684 0.6905 0.4248 0.092 Uiso 1 1 calc R . . . .

C21 C 0.5168(3) 0.8465(4) 0.4306(3) 0.1071(16) Uani 1 1 d . . . . .

H21A H 0.4952 0.9152 0.3997 0.161 Uiso 1 1 calc GR . . . .

H21B H 0.5760 0.8247 0.4136 0.161 Uiso 1 1 calc GR . . . .

H21C H 0.5274 0.8554 0.4987 0.161 Uiso 1 1 calc GR . . . .

loop_

_atom_site_aniso_label

_atom_site_aniso_U_11

_atom_site_aniso_U_22

_atom_site_aniso_U_33

_atom_site_aniso_U_23

_atom_site_aniso_U_13

_atom_site_aniso_U_12

P1 0.0654(5) 0.0412(4) 0.0421(4) 0.0014(3) 0.0124(3) -0.0174(4)

O1 0.1067(19) 0.0669(15) 0.0464(11) 0.0080(11) 0.0183(12) -0.0346(13)

O2 0.0847(16) 0.0381(11) 0.0639(13) -0.0016(10) 0.0134(11) -0.0063(10)

O3 0.0562(12) 0.0575(13) 0.0515(11) -0.0092(9) 0.0144(9) -0.0176(10)

O4 0.0537(11) 0.0456(11) 0.0399(9) -0.0094(8) 0.0188(8) -0.0153(9)

O5 0.123(2) 0.0770(18) 0.0593(14) 0.0070(13) 0.0244(14) 0.0172(16)

N1 0.0509(13) 0.0394(13) 0.0447(12) 0.0030(10) 0.0165(10) -0.0118(10)

C1 0.0500(16) 0.0407(15) 0.0550(16) 0.0028(13) 0.0143(13) -0.0078(12)

C2 0.071(2) 0.0518(18) 0.0387(14) -0.0107(13) 0.0131(13) -0.0128(15)

C3 0.0609(18) 0.0522(17) 0.0403(14) -0.0152(13) 0.0195(13) -0.0146(14)

C4 0.059(2) 0.070(2) 0.070(2) -0.0229(17) 0.0177(16) -0.0153(17)

C5 0.061(2) 0.090(3) 0.085(2) -0.014(2) 0.0227(18) 0.005(2)

C6 0.093(3) 0.065(2) 0.082(2) -0.0088(19) 0.036(2) 0.009(2)

C7 0.093(3) 0.052(2) 0.067(2) -0.0200(17) 0.0294(19) -0.0190(19)

C8 0.0607(18) 0.0575(19) 0.0502(16) -0.0136(14) 0.0155(14) -0.0132(15)

C9 0.157(4) 0.069(2) 0.0508(19) -0.0067(17) 0.049(2) -0.016(2)

C10 0.0648(18) 0.0348(14) 0.0439(14) 0.0021(12) 0.0220(13) -0.0066(13)

C11 0.082(2) 0.059(2) 0.0404(15) 0.0065(14) 0.0155(15) -0.0019(16)

C12 0.140(4) 0.0384(18) 0.0558(19) 0.0028(14) 0.032(2) -0.0034(19)

C13 0.083(3) 0.092(3) 0.080(2) 0.025(2) 0.043(2) -0.007(2)

C14 0.0503(18) 0.060(2) 0.087(2) 0.0133(19) 0.0022(16) -0.0060(16)

C15 0.082(3) 0.112(4) 0.075(3) 0.006(2) -0.019(2) -0.009(2)

C16 0.070(3) 0.074(3) 0.155(4) 0.021(3) -0.010(3) 0.012(2)

C17 0.054(2) 0.085(3) 0.136(4) 0.011(3) 0.007(2) -0.0180(19)

C18 0.167(5) 0.041(2) 0.114(3) 0.000(2) 0.020(3) -0.024(3)

C19 0.205(6) 0.054(3) 0.124(4) -0.018(3) -0.033(4) 0.036(3)

C20 0.061(2) 0.085(3) 0.090(2) -0.022(2) 0.0275(19) -0.0205(19)

C21 0.082(3) 0.139(4) 0.107(3) -0.038(3) 0.035(2) -0.057(3)

_geom_special_details

All esds (except the esd in the dihedral angle between two l.s. planes)

are estimated using the full covariance matrix. The cell esds are taken

into account individually in the estimation of esds in distances, angles

and torsion angles; correlations between esds in cell parameters are only

used when they are defined by crystal symmetry. An approximate (isotropic)

treatment of cell esds is used for estimating esds involving l.s. planes.

loop_

_geom_bond_atom_site_label_1

_geom_bond_atom_site_label_2

_geom_bond_distance

_geom_bond_site_symmetry_2

_geom_bond_publ_flag

P1 O1 1.463(2) . ?

P1 O2 1.577(2) . ?

P1 O3 1.563(2) . ?

P1 C1 1.835(3) . ?

O2 C18 1.435(4) . ?

O3 C20 1.448(4) . ?

O4 N1 1.458(3) . ?

O4 C2 1.451(3) . ?

O5 H5 0.8200 . ?

O5 C11 1.423(4) . ?

N1 C1 1.488(3) . ?

N1 C10 1.513(3) . ?

C1 H1 0.9800 . ?

C1 C14 1.586(4) . ?

C2 H2 0.9800 . ?

C2 C3 1.510(4) . ?

C2 C9 1.521(4) . ?

C3 C4 1.384(4) . ?

C3 C8 1.387(4) . ?

C4 H4 0.9300 . ?

C4 C5 1.378(5) . ?

C5 H5A 0.9300 . ?

C5 C6 1.374(5) . ?

C6 H6 0.9300 . ?

C6 C7 1.364(5) . ?

C7 H7 0.9300 . ?

C7 C8 1.373(5) . ?

C8 H8 0.9300 . ?

C9 H9A 0.9600 . ?

C9 H9B 0.9600 . ?

C9 H9C 0.9600 . ?

C10 C11 1.512(4) . ?

C10 C12 1.517(4) . ?

C10 C13 1.523(4) . ?

C11 H11A 0.9700 . ?

C11 H11B 0.9700 . ?

C12 H12A 0.9600 . ?

C12 H12B 0.9600 . ?

C12 H12C 0.9600 . ?

C13 H13A 0.9600 . ?

C13 H13B 0.9600 . ?

C13 H13C 0.9600 . ?

C14 C15 1.538(5) . ?

C14 C16 1.533(6) . ?

C14 C17 1.522(5) . ?

C15 H15A 0.9600 . ?

C15 H15B 0.9600 . ?

C15 H15C 0.9600 . ?

C16 H16A 0.9600 . ?

C16 H16B 0.9600 . ?

C16 H16C 0.9600 . ?

C17 H17A 0.9600 . ?

C17 H17B 0.9600 . ?

C17 H17C 0.9600 . ?

C18 H18A 0.9700 . ?

C18 H18B 0.9700 . ?

C18 C19 1.441(7) . ?

C19 H19A 0.9600 . ?

C19 H19B 0.9600 . ?

C19 H19C 0.9600 . ?

C20 H20A 0.9700 . ?

C20 H20B 0.9700 . ?

C20 C21 1.462(5) . ?

C21 H21A 0.9600 . ?

C21 H21B 0.9600 . ?

C21 H21C 0.9600 . ?

loop_

_geom_angle_atom_site_label_1

_geom_angle_atom_site_label_2

_geom_angle_atom_site_label_3

_geom_angle

_geom_angle_site_symmetry_1

_geom_angle_site_symmetry_3

_geom_angle_publ_flag

O1 P1 O2 114.03(13) . . ?

O1 P1 O3 113.85(14) . . ?

O1 P1 C1 116.38(13) . . ?

O2 P1 C1 98.45(13) . . ?

O3 P1 O2 101.26(11) . . ?

O3 P1 C1 110.88(12) . . ?

C18 O2 P1 120.2(3) . . ?

C20 O3 P1 123.9(2) . . ?

C2 O4 N1 113.39(19) . . ?

C11 O5 H5 109.5 . . ?

O4 N1 C1 107.04(18) . . ?

O4 N1 C10 108.12(19) . . ?

C1 N1 C10 116.6(2) . . ?

P1 C1 H1 105.3 . . ?

N1 C1 P1 115.76(19) . . ?

N1 C1 H1 105.3 . . ?

N1 C1 C14 111.6(2) . . ?

C14 C1 P1 112.6(2) . . ?

C14 C1 H1 105.3 . . ?

O4 C2 H2 109.0 . . ?

O4 C2 C3 113.1(2) . . ?

O4 C2 C9 106.6(2) . . ?

C3 C2 H2 109.0 . . ?

C3 C2 C9 110.1(3) . . ?

C9 C2 H2 109.0 . . ?

C4 C3 C2 122.3(3) . . ?

C4 C3 C8 117.7(3) . . ?

C8 C3 C2 120.0(3) . . ?

C3 C4 H4 119.6 . . ?

C5 C4 C3 120.7(3) . . ?

C5 C4 H4 119.6 . . ?

C4 C5 H5A 119.7 . . ?

C6 C5 C4 120.6(4) . . ?

C6 C5 H5A 119.7 . . ?

C5 C6 H6 120.4 . . ?

C7 C6 C5 119.3(4) . . ?

C7 C6 H6 120.4 . . ?

C6 C7 H7 119.8 . . ?

C6 C7 C8 120.5(3) . . ?

C8 C7 H7 119.8 . . ?

C3 C8 H8 119.4 . . ?

C7 C8 C3 121.2(3) . . ?

C7 C8 H8 119.4 . . ?

C2 C9 H9A 109.5 . . ?

C2 C9 H9B 109.5 . . ?

C2 C9 H9C 109.5 . . ?

H9A C9 H9B 109.5 . . ?

H9A C9 H9C 109.5 . . ?

H9B C9 H9C 109.5 . . ?

N1 C10 C12 107.2(2) . . ?

N1 C10 C13 109.3(2) . . ?

C11 C10 N1 113.6(2) . . ?

C11 C10 C12 110.5(3) . . ?

C11 C10 C13 108.9(3) . . ?

C12 C10 C13 107.1(3) . . ?

O5 C11 C10 111.3(3) . . ?

O5 C11 H11A 109.4 . . ?

O5 C11 H11B 109.4 . . ?

C10 C11 H11A 109.4 . . ?

C10 C11 H11B 109.4 . . ?

H11A C11 H11B 108.0 . . ?

C10 C12 H12A 109.5 . . ?

C10 C12 H12B 109.5 . . ?

C10 C12 H12C 109.5 . . ?

H12A C12 H12B 109.5 . . ?

H12A C12 H12C 109.5 . . ?

H12B C12 H12C 109.5 . . ?

C10 C13 H13A 109.5 . . ?

C10 C13 H13B 109.5 . . ?

C10 C13 H13C 109.5 . . ?

H13A C13 H13B 109.5 . . ?

H13A C13 H13C 109.5 . . ?

H13B C13 H13C 109.5 . . ?

C15 C14 C1 112.6(3) . . ?

C16 C14 C1 111.0(3) . . ?

C16 C14 C15 108.4(3) . . ?

C17 C14 C1 108.9(3) . . ?

C17 C14 C15 108.1(3) . . ?

C17 C14 C16 107.6(3) . . ?

C14 C15 H15A 109.5 . . ?

C14 C15 H15B 109.5 . . ?

C14 C15 H15C 109.5 . . ?

H15A C15 H15B 109.5 . . ?

H15A C15 H15C 109.5 . . ?

H15B C15 H15C 109.5 . . ?

C14 C16 H16A 109.5 . . ?

C14 C16 H16B 109.5 . . ?

C14 C16 H16C 109.5 . . ?

H16A C16 H16B 109.5 . . ?

H16A C16 H16C 109.5 . . ?

H16B C16 H16C 109.5 . . ?

C14 C17 H17A 109.5 . . ?

C14 C17 H17B 109.5 . . ?

C14 C17 H17C 109.5 . . ?

H17A C17 H17B 109.5 . . ?

H17A C17 H17C 109.5 . . ?

H17B C17 H17C 109.5 . . ?

O2 C18 H18A 109.6 . . ?

O2 C18 H18B 109.6 . . ?

O2 C18 C19 110.5(5) . . ?

H18A C18 H18B 108.1 . . ?

C19 C18 H18A 109.6 . . ?

C19 C18 H18B 109.6 . . ?

C18 C19 H19A 109.5 . . ?

C18 C19 H19B 109.5 . . ?

C18 C19 H19C 109.5 . . ?

H19A C19 H19B 109.5 . . ?

H19A C19 H19C 109.5 . . ?

H19B C19 H19C 109.5 . . ?

O3 C20 H20A 109.5 . . ?

O3 C20 H20B 109.5 . . ?

O3 C20 C21 110.7(3) . . ?

H20A C20 H20B 108.1 . . ?

C21 C20 H20A 109.5 . . ?

C21 C20 H20B 109.5 . . ?

C20 C21 H21A 109.5 . . ?

C20 C21 H21B 109.5 . . ?

C20 C21 H21C 109.5 . . ?

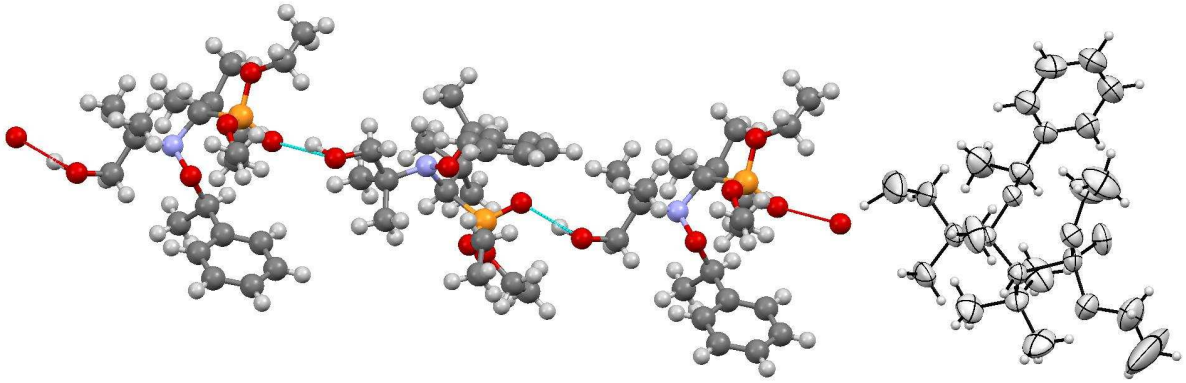
H21A C21 H21B 109.5 . . ?

H21A C21 H21C 109.5 . . ?

H21B C21 H21C 109.5 . . ?

_olex2_submission_special_instructions 'No special instructions were received'

(*RS/SR*)-2



_publ_section_comment

;

The study of the titled structure was undertaken to establish its three

dimensional structure. Geometries are tabulated below. All Diagrams and

calculations were performed using maXus (MacScience, Japan).

;

_chemical_compound_source 'Local laboratory'

_exptl_crystal_description 'prism'

_exptl_crystal_colour 'Colourless'

_exptl_crystal_size_max 0.5

_exptl_crystal_size_mid 0.5

_exptl_crystal_size_min 0.4

_chemical_formula_sum 'C21 H36 P1 O5 N1'

_chemical_formula_weight 413.49

_cell_length_a 9.0756(3)

_cell_length_b 16.0249(8)

_cell_length_c 16.9442(8)

_cell_angle_alpha 90.00(1)

_cell_angle_beta 100.193(2)

_cell_angle_gamma 90.00(1)

_cell_volume 2425.4(3)

_cell_measurement_reflns_used '16552'

_cell_measurement_theta_min '1'

_cell_measurement_theta_max '25'

_cell_formula_units_Z 5

_exptl_crystal_density_diffrn 1.42

_exptl_crystal_density_meas 1.25

_exptl_absorpt_coefficient_mu 1.41

_cell_measurement_temperature '298'

_exptl_absorpt_correction_type 'none'

_diffrn_reflns_number 4804

_reflns_number_total 4614

_reflns_number_observed 3678

_reflns_observed_criterion 'refl_observed_if_I_>_3.00_sigma(I)'

_diffrn_reflns_theta_max 25.41

_refine_ls_structure_factor_coef F

_refine_ls_R_factor_obs 0.062

_refine_ls_wR_factor_obs 0.095

_refine_ls_hydrogen_treatment 'noref'

_refine_ls_number_reflns 3678

_refine_ls_number_parameters 253

_refine_ls_goodness_of_fit_obs 1.996

_refine_ls_weighting_scheme 'Count statistics'

_refine_ls_shift/esd_max 0.0276

_refine_ls_shift/esd_mean 0.0042

_refine_diff_density_min -0.26

_refine_diff_density_max 0.46

_refine_ls_extinction_method 'None'

_atom_type_scat_source 'D.Waasmaier&A.Kirfel,Acta Cryst.1995,A51, 416-431'

_computing_data_reduction 'maXus'

_computing_molecular_graphics 'maXus'

_computing_publication_material 'maXus'

_computing_structure_refinement 'maXus'

_computing_structure_solution 'maXus'

_diffrn_measurement_device 'KappaCCD'

_computing_data_collection 'KappaCCD'

_diffrn_radiation_type 'xray'

_diffrn_reflns_limit_h_min 0

_diffrn_reflns_limit_h_max 10

_diffrn_reflns_limit_k_min 0

_diffrn_reflns_limit_k_max 19

_diffrn_reflns_limit_l_min -21

_diffrn_reflns_limit_l_max 20

_diffrn_radiation_wavelength 0.71073

_diffrn_orient_matrix_type 'X=UH'

_diffrn_orient_matrix_UB_11 0.01000

_diffrn_orient_matrix_UB_12 0.00000

_diffrn_orient_matrix_UB_13 0.00000

_diffrn_orient_matrix_UB_21 0.00000

_diffrn_orient_matrix_UB_22 0.01000

_diffrn_orient_matrix_UB_23 0.00000

_diffrn_orient_matrix_UB_31 0.00000

_diffrn_orient_matrix_UB_32 0.00000

_diffrn_orient_matrix_UB_33 0.01000

loop_

_symmetry_equiv_pos_as_xyz

+X,+Y,+Z

-X,-Y,-Z

-X+0.5,+Y+0.5,-Z+0.5

+X+0.5,-Y+0.5,+Z+0.5

_symmetry_space_group_name_H-M 'P 21/n '

_symmetry_cell_setting 'Monoclinic'

#==========

ATOMIC COORDINATES AND THERMAL PARAMETERS

loop_

_atom_site_label

_atom_site_fract_x

_atom_site_fract_y

_atom_site_fract_z

_atom_site_U_iso_or_equiv

_atom_site_occupancy

_atom_site_thermal_displace_type

P1	0.98620(3)	0.16370(2)	0.31940(1)	0.0510(1)	1.00 Uij
O2	0.83870(6)	0.30080(4)	0.25110(3)	0.0481(3)	1.00 Uij
N3	0.89990(7)	0.26500(5)	0.18460(4)	0.0457(4)	1.00 Uij
O4	0.82050(7)	0.14470(4)	0.32720(4)	0.0586(4)	1.00 Uij
O5	1.06440(8)	0.21530(5)	0.38480(4)	0.0680(4)	1.00 Uij
O6	1.04930(8)	0.07200(5)	0.31670(5)	0.0718(4)	1.00 Uij
C7	1.0026(1)	0.19550(6)	0.21680(5)	0.0481(5)	1.00 Uij
C8	0.9135(1)	0.37720(6)	0.28240(6)	0.0586(5)	1.00 Uij

C9	0.7641(1)	0.23840(7)	0.12390(5)	0.0557(5)	1.00 Uij
C10	0.8175(1)	0.20340(8)	0.04980(6)	0.0735(6)	1.00 Uij
C11	0.8270(1)	0.40380(6)	0.34680(6)	0.0575(5)	1.00 Uij
C12	1.1699(1)	0.21340(7)	0.20540(6)	0.0640(6)	1.00 Uij
C13	0.8975(1)	0.40970(8)	0.42540(7)	0.0724(7)	1.00 Uij
C14	0.6764(1)	0.31820(9)	0.09840(7)	0.0861(8)	1.00 Uij
C15	1.2485(1)	0.2826(1)	0.25900(9)	0.1006(9)	1.00 Uij
O16	0.5456(1)	0.30740(9)	0.04420(6)	0.1249(7)	1.00 Uij
C17	0.6763(1)	0.42240(9)	0.32860(7)	0.0831(8)	1.00 Uij
C18	0.7366(2)	0.19230(9)	0.37780(9)	0.0911(8)	1.00 Uij
C19	1.1716(1)	0.2400(1)	0.11880(8)	0.0851(8)	1.00 Uij
C20	0.6645(1)	0.1746(1)	0.15510(7)	0.0872(8)	1.00 Uij
C21	0.5973(2)	0.4446(1)	0.3890(1)	0.103(1)	1.00 Uij
C22	0.8187(2)	0.43330(9)	0.48490(8)	0.0911(9)	1.00 Uij
C23	0.9166(2)	0.44350(8)	0.21940(8)	0.0896(8)	1.00 Uij
C24	1.0694(2)	0.02020(9)	0.38800(9)	0.103(1)	1.00 Uij
C25	1.2634(1)	0.1333(1)	0.2197(1)	0.1045(9)	1.00 Uij
C26	0.6226(2)	0.1440(1)	0.4032(1)	0.125(1)	1.00 Uij
C27	0.6712(2)	0.44950(9)	0.46690(9)	0.098(1)	1.00 Uij

C28	1.1979(4)	-0.0219(3)	0.3978(2)	0.336(5)	1.00 Uij
H7	0.97190	0.15030	0.18040	0.08730	1.00 Uiso
H8	1.01620	0.36510	0.30430	0.09810	1.00 Uiso
H10A	0.87970	0.24350	0.02940	0.11230	1.00 Uiso
H10B	0.73230	0.19090	0.00930	0.11230	1.00 Uiso
H10C	0.87370	0.15330	0.06440	0.11230	1.00 Uiso
H13	1.00300	0.39880	0.43890	0.11120	1.00 Uiso
H14A	0.65070	0.34530	0.14470	0.12380	1.00 Uiso
H14B	0.73730	0.35480	0.07290	0.12380	1.00 Uiso
H15A	1.19070	0.33300	0.25000	0.14010	1.00 Uiso
H15B	1.34660	0.29220	0.24700	0.14010	1.00 Uiso
H15C	1.25680	0.26610	0.31410	0.14010	1.00 Uiso
H17	0.62540	0.41950	0.27390	0.12200	1.00 Uiso
H18A	0.80470	0.21240	0.42370	0.13240	1.00 Uiso
H18B	0.68930	0.23880	0.34770	0.13240	1.00 Uiso
H19A	1.11530	0.29070	0.10780	0.12550	1.00 Uiso
H19B	1.12710	0.19700	0.08280	0.12550	1.00 Uiso
H19C	1.27280	0.24920	0.11150	0.12550	1.00 Uiso
H20A	0.72210	0.12520	0.17080	0.12550	1.00 Uiso

H20B	0.58140	0.16100	0.11370	0.12550	1.00 Uiso
H20C	0.62800	0.19690	0.20060	0.12550	1.00 Uiso
H21	0.49220	0.45660	0.37620	0.14240	1.00 Uiso
H22	0.86930	0.43790	0.53950	0.13040	1.00 Uiso
H23A	0.97220	0.42460	0.17960	0.12950	1.00 Uiso
H23B	0.96260	0.49290	0.24470	0.12950	1.00 Uiso
H23C	0.81560	0.45590	0.19430	0.12950	1.00 Uiso
H24A	1.07450	0.05600	0.43380	0.14170	1.00 Uiso
H24B	0.98640	-0.01740	0.38570	0.14170	1.00 Uiso
H25A	1.26560	0.11430	0.27360	0.14460	1.00 Uiso
H25B	1.36370	0.14360	0.21140	0.14460	1.00 Uiso
H25C	1.21800	0.09140	0.18270	0.14460	1.00 Uiso
H26A	0.56810	0.17590	0.43640	0.16720	1.00 Uiso
H26B	0.67020	0.09750	0.43320	0.16720	1.00 Uiso
H26C	0.55480	0.12390	0.35710	0.16720	1.00 Uiso
H27	0.61600	0.46420	0.50830	0.13840	1.00 Uiso
H28A	1.21320	-0.05550	0.44540	0.38020	1.00 Uiso
H28B	1.28010	0.01620	0.39970	0.38020	1.00 Uiso
H28C	1.19200	-0.05720	0.35160	0.38020	1.00 Uiso

H16 0.57420 0.30940 -0.01860 0.16250 1.00 Uiso

loop_

_atom_site_aniso_label

_atom_site_aniso_U_11

_atom_site_aniso_U_22

_atom_site_aniso_U_33

_atom_site_aniso_U_12

_atom_site_aniso_U_13

_atom_site_aniso_U_23

P1 0.0518(2) 0.0597(2) 0.0386(1) -.0001(1) 0.00020(9) 0.0030(1)

O2 0.0527(4) 0.0543(4) 0.0361(3) 0.0020(3) 0.0096(3) -.0032(3)

N3 0.0435(4) 0.0609(5) 0.0312(4) 0.0053(3) 0.0051(3) -.0017(3)

O4 0.0592(4) 0.0639(4) 0.0520(4) -.0063(3) 0.0152(3) -.0019(3)

O5 0.0761(5) 0.0833(5) 0.0398(4) -.0157(4) -.0060(3) -.0005(3)

O6 0.0792(5) 0.0666(5) 0.0666(5) 0.0152(4) 0.0097(4) 0.0139(4)

C7 0.0431(5) 0.0607(6) 0.0382(5) 0.0043(4) 0.0024(3) -.0005(4)

C8 0.0634(6) 0.0534(6) 0.0574(6) -.0067(5) 0.0126(5) -.0065(5)

C9	0.0493(5)	0.0807(7)	0.0342(5)	0.0077(5)	-.0013(4)	-.0052(5)
C10	0.0711(7)	0.1026(9)	0.0429(6)	0.0122(6)	-.0009(5)	-.0184(6)
C11	0.0693(7)	0.0478(6)	0.0532(6)	0.0032(5)	0.0092(5)	-.0057(5)
C12	0.0431(5)	0.0890(8)	0.0588(6)	0.0056(5)	0.0113(4)	0.0060(6)
C13	0.0817(8)	0.0732(8)	0.0587(7)	0.0073(6)	0.0059(6)	-.0056(6)
C14	0.0861(8)	0.118(1)	0.0469(6)	0.0490(7)	-.0161(5)	-.0079(6)
C15	0.0590(8)	0.149(1)	0.092(1)	-.0325(8)	0.0198(6)	-.0201(9)
O16	0.0979(7)	0.209(1)	0.0606(6)	0.0584(7)	-.0113(5)	-.0085(6)
C17	0.0821(9)	0.100(1)	0.0641(7)	0.0266(7)	0.0068(6)	-.0139(7)
C18	0.0886(9)	0.0926(9)	0.096(1)	-.0118(7)	0.0534(7)	-.0214(8)
C19	0.0684(8)	0.115(1)	0.0733(8)	0.0020(7)	0.0311(6)	0.0125(7)
C20	0.0543(7)	0.146(1)	0.0561(7)	-.0293(7)	-.0092(5)	-.0040(7)
C21	0.089(1)	0.120(1)	0.098(1)	0.0407(8)	0.0233(8)	-.0126(9)
C22	0.115(1)	0.099(1)	0.0573(7)	0.0207(8)	0.0168(7)	-.0104(7)
C23	0.115(1)	0.0692(8)	0.0842(9)	-.0160(7)	0.0337(8)	0.0047(7)
C24	0.127(1)	0.081(1)	0.097(1)	0.0135(8)	0.0142(9)	0.0370(8)
C25	0.0589(8)	0.128(1)	0.126(1)	0.0311(8)	0.0336(7)	0.027(1)
C26	0.121(1)	0.141(1)	0.120(1)	-.021(1)	0.073(1)	-.021(1)
C27	0.125(1)	0.098(1)	0.0722(9)	0.0339(9)	0.0347(8)	-.0061(8)

C28 0.309(4) 0.401(5) 0.308(4) 0.273(4) 0.169(3) 0.265(4)

loop_

_geom_bond_atom_site_label_1

_geom_bond_atom_site_label_2

_geom_bond_distance

_geom_bond_site_symmetry_1

_geom_bond_site_symmetry_2

_geom_bond_publ_flag

P1 O4 1.563(1) . . ?

P1 O5 1.462(1) . . ?

P1 O6 1.581(1) . . ?

P1 C7 1.843(1) . . ?

O2 N3 1.459(1) . . ?

O2 C8 1.454(2) . . ?

N3 C7 1.492(2) . . ?

N3 C9 1.520(2) . . ?

O4 C18 1.459(2) . . ?

O6 C24 1.450(2) . . ?

C7	C12	1.590(2) . . ?	
C8	C11	1.514(2) . . ?	
C8	C23	1.510(2) . . ?	
C9	C10	1.530(2) . . ?	
C9	C14	1.528(2) . . ?	
C9	C20	1.520(2) . . ?	
C11	C13	1.375(2) . . ?	
C11	C17	1.380(2) . . ?	
C12	C15	1.528(2) . . ?	
C12	C19	1.531(2) . . ?	
C12	C25	1.534(2) . . ?	
C13	C22	1.388(2) . . ?	
C14	O16	1.377(2) . . ?	
C17	C21	1.396(3) . . ?	
C18	C26	1.419(3) . . ?	
C21	C27	1.373(3) . . ?	
C22	C27	1.345(3) . . ?	
C24	C28	1.332(5) . . ?	
O5	H16	1.671(1) .	4_656 ?

O16 H14A 1.898(2) . . ?

O16 H14B 1.882(2) . . ?

O16 H16 1.139(2) . . ?

C24 H28B 1.888(2) . . ?

C24 H28C 1.844(2) . . ?

C28 H24A 1.851(5) . . ?

C28 H24B 1.895(4) . . ?

loop_

_geom_angle_atom_site_label_1

_geom_angle_atom_site_label_2

_geom_angle_atom_site_label_3

_geom_angle

_geom_angle_site_symmetry_1

_geom_angle_site_symmetry_2

_geom_angle_site_symmetry_3

_geom_angle_publ_flag

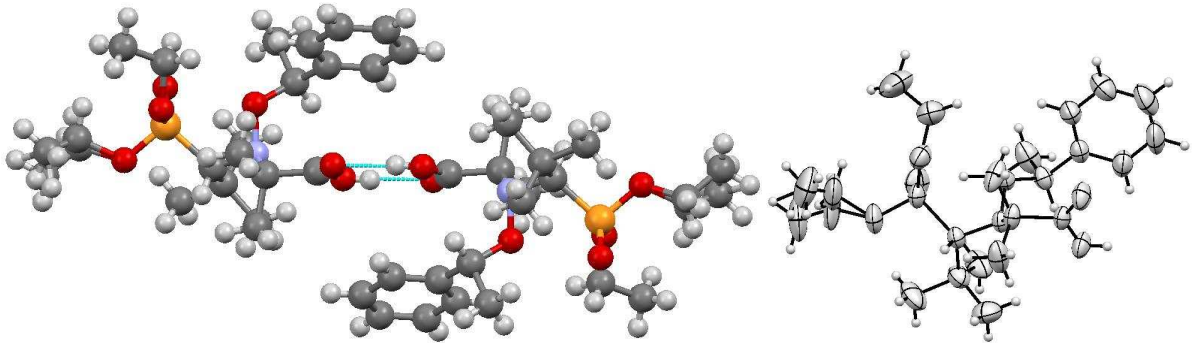
O4 P1 O5 113.0(1) . . . ?

O4	P1	O6	100.4(1)	. . . ?
O4	P1	C7	111.9(1)	. . . ?
O5	P1	O6	114.7(1)	. . . ?
O5	P1	C7	116.7(1)	. . . ?
O6	P1	C7	98.0(1)	. . . ?
N3	O2	C8	113.3(1)	. . . ?
O2	N3	C7	108.0(1)	. . . ?
O2	N3	C9	105.0(1)	. . . ?
C7	N3	C9	114.7(1)	. . . ?
P1	O4	C18	123.9(1)	. . . ?
P1	O6	C24	120.0(1)	. . . ?
P1	C7	N3	113.7(1)	. . . ?
P1	C7	C12	113.7(1)	. . . ?
N3	C7	C12	111.7(1)	. . . ?
O2	C8	C11	103.4(1)	. . . ?
O2	C8	C23	113.6(1)	. . . ?
C11	C8	C23	112.9(1)	. . . ?
N3	C9	C10	108.7(1)	. . . ?
N3	C9	C14	106.0(1)	. . . ?

N3	C9	C20	114.5(1)	. . . ?
C10	C9	C14	107.4(1)	. . . ?
C10	C9	C20	109.5(1)	. . . ?
C14	C9	C20	110.4(1)	. . . ?
C8	C11	C13	120.4(1)	. . . ?
C8	C11	C17	121.3(1)	. . . ?
C13	C11	C17	118.3(1)	. . . ?
C7	C12	C15	114.5(1)	. . . ?
C7	C12	C19	109.8(1)	. . . ?
C7	C12	C25	110.1(1)	. . . ?
C15	C12	C19	106.6(2)	. . . ?
C15	C12	C25	109.0(1)	. . . ?
C19	C12	C25	106.6(2)	. . . ?
C11	C13	C22	120.7(2)	. . . ?
C9	C14	O16	115.3(2)	. . . ?
C11	C17	C21	120.6(2)	. . . ?
O4	C18	C26	112.2(2)	. . . ?
C17	C21	C27	119.5(2)	. . . ?
C13	C22	C27	120.5(2)	. . . ?

O6	C24	C28	111.8(3)	. . .	?
C21	C27	C22	120.3(2)	. . .	?
P1	O5	H16	122.9(1)	. .	4_656 ?
C14	O16	H14A	28.9(1)	. . .	?
C14	O16	H14B	29.4(1)	. . .	?
C14	O16	H16	107.9(1)	. . .	?
H14A	O16	H14B	49.0(1)	. . .	?
H14A	O16	H16	131.9(1)	. . .	?
H14B	O16	H16	82.9(1)	. . .	?
O6	C24	H28B	94.7(1)	. . .	?
O6	C24	H28C	95.8(1)	. . .	?
C28	C24	H28B	28.6(2)	. . .	?
C28	C24	H28C	30.0(2)	. . .	?
H28B	C24	H28C	49.6(1)	. . .	?
C24	C28	H24A	29.8(2)	. . .	?
C24	C28	H24B	28.3(2)	. . .	?
H24A	C28	H24B	49.4(2)	. . .	?
O5	H16	O16	157.9(1)	4_555	. . ?

(*RR/SS*)-4



Olex2 1.2

(compiled 2015.09.30 svn.r3233 for OlexSys, GUI svn.r5103)

;

_shelxl_version_number 2014/6

_publ_contact_author_address ?

_publ_contact_author_email ?

_publ_contact_author_name ''

_publ_contact_author_phone ?

_publ_section_references

;

Dolomanov, O.V., Bourhis, L.J., Gildea, R.J, Howard, J.A.K. & Puschmann, H.

(2009), J. Appl. Cryst. 42, 339-341.

Sheldrick, G.M. (2008). Acta Cryst. A64, 112-122.

Sheldrick, G.M. (2015). Acta Cryst. C71, 3-8.

;

_chemical_name_common ?

_chemical_name_systematic ?

_chemical_formula_moiety 'C21 H36 N O6 P'

_chemical_formula_sum 'C21 H36 N O6 P'

_chemical_formula_weight 429.48

_chemical_melting_point ?

loop_

_atom_type_symbol

_atom_type_description

_atom_type_scat_dispersion_real

_atom_type_scat_dispersion_imag

_atom_type_scat_source

'C' 'C' 0.0181 0.0091 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

'H' 'H' 0.0000 0.0000 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

'N' 'N' 0.0311 0.0180 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

'O' 'O' 0.0492 0.0322 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

'P' 'P' 0.2955 0.4335 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

_shelx_space_group_comment

;

The symmetry employed for this shelxl refinement is uniquely defined

by the following loop, which should always be used as a source of

symmetry information in preference to the above space-group names.

They are only intended as comments.

;

_space_group_crystal_system 'monoclinic'

_space_group_IT_number 14

_space_group_name_H-M_alt 'P 1 21/n 1'

_space_group_name_Hall '-P 2yn'

loop_

_space_group_symop_operation_xyz

'x, y, z'

'-x+1/2, y+1/2, -z+1/2'

'-x, -y, -z'

'x-1/2, -y-1/2, z-1/2'

_cell_length_a	15.9524(3)
_cell_length_b	8.81848(11)
_cell_length_c	17.6457(2)
_cell_angle_alpha	90
_cell_angle_beta	104.1340(15)
_cell_angle_gamma	90
_cell_volume	2407.18(6)
_cell_formula_units_Z	4
_cell_measurement_reflns_used	9677
_cell_measurement_temperature	293
_cell_measurement_theta_max	74.3210
_cell_measurement_theta_min	5.1620
_shelx_estimated_absorpt_T_max	?
_shelx_estimated_absorpt_T_min	?
_exptl_absorpt_coefficient_mu	1.294
_exptl_absorpt_correction_T_max	1.00000
_exptl_absorpt_correction_T_min	0.78044

_exptl_absorpt_correction_type multi-scan

_exptl_absorpt_process_details

;

CrysAlisPro, Agilent Technologies,

Version 1.171.37.35 (release 13-08-2014 CrysAlis171 .NET)

(compiled Aug 13 2014,18:06:01)

Empirical absorption correction using spherical harmonics,

implemented in SCALE3 ABSPACK scaling algorithm.

;

_exptl_absorpt_special_details ?

_exptl_crystal_colour colourless

_exptl_crystal_colour_primary colourless

_exptl_crystal_density_diffrn 1.185

_exptl_crystal_density_meas ?

_exptl_crystal_density_method ?

_exptl_crystal_description prism

_exptl_crystal_F_000 928

_exptl_crystal_size_max 0.28

_exptl_crystal_size_mid 0.16

_exptl_crystal_size_min 0.1

_exptl_transmission_factor_max ?

_exptl_transmission_factor_min ?

_diffrn_reflns_av_R_equivalents 0.0265

_diffrn_reflns_av_unetI/netI 0.0231

_diffrn_reflns_Laue_measured_fraction_full 0.999

_diffrn_reflns_Laue_measured_fraction_max 0.990

_diffrn_reflns_limit_h_max 19

_diffrn_reflns_limit_h_min -19

_diffrn_reflns_limit_k_max 10

_diffrn_reflns_limit_k_min -10

_diffrn_reflns_limit_l_max 21

_diffrn_reflns_limit_l_min -21

_diffrn_reflns_number 18665

_diffrn_reflns_point_group_measured_fraction_full 0.999

_diffrn_reflns_point_group_measured_fraction_max 0.990

_diffrn_reflns_theta_full 67.684

_diffrn_reflns_theta_max 74.350

_diffrn_reflns_theta_min 4.295

```
_diffrn_ambient_temperature             293
_diffrn_detector                        'CCD plate'
_diffrn_detector_area_resol_mean    5.3048
_diffrn_detector_type                   AtlasS2
_diffrn_measured_fraction_theta_full 0.999
_diffrn_measured_fraction_theta_max 0.990
_diffrn_measurement_details
;
#__ type_ start__ end____ width___ exp.time_
   1 omega    -14.00      12.00      1.0000        1.0000
omega____ theta____ kappa____ phi______ frames
       -              0.0000    -84.0000      69.0000 26


#__ type_ start__ end____ width___ exp.time_
   2 omega      14.00      41.00      1.0000        1.0000
omega____ theta____ kappa____ phi______ frames
       -              0.0000    -99.0000     120.0000 27


#__ type_ start__ end____ width___ exp.time_
```

3 omega -34.00 27.00 1.0000 1.0000

omega____ theta____ kappa____ phi_____ frames

- -57.0245 38.0000 150.0000 61

#__ type_ start__ end____ width___ exp.time_

4 omega -85.00 -23.00 1.0000 1.0000

omega____ theta____ kappa____ phi_____ frames

- -57.0245 -57.0000 90.0000 62

#__ type_ start__ end____ width___ exp.time_

5 omega 1.00 26.00 1.0000 1.0000

omega____ theta____ kappa____ phi_____ frames

- 0.0000 -99.0000 -90.0000 25

#__ type_ start__ end____ width___ exp.time_

6 omega 32.00 88.00 1.0000 8.0000

omega____ theta____ kappa____ phi_____ frames

- 114.0489 -30.0000 90.0000 56

#__ type_ start__ end____ width___ exp.time_

7 omega 57.00 83.00 1.0000 8.0000

omega____ theta____ kappa____ phi_____ frames

- 114.0489 -45.0000 30.0000 26

#__ type_ start__ end____ width___ exp.time_

8 omega 58.00 89.00 1.0000 8.0000

omega____ theta____ kappa____ phi_____ frames

- 114.0489 -30.0000 30.0000 31

#__ type_ start__ end____ width___ exp.time_

9 omega 80.00 107.00 1.0000 8.0000

omega____ theta____ kappa____ phi_____ frames

- 114.0489 45.0000 120.0000 27

#__ type_ start__ end____ width___ exp.time_

10 omega 84.00 162.00 1.0000 8.0000

omega____ theta____ kappa____ phi_____ frames

- 114.0489 45.0000 150.0000 78

#__ type_ start__ end____ width___ exp.time_

11 omega 153.00 178.00 1.0000 8.0000

omega____ theta____ kappa____ phi_____ frames

- 114.0489 61.0000 120.0000 25

#__ type_ start__ end____ width___ exp.time_

12 omega 112.00 138.00 1.0000 1.0000

omega____ theta____ kappa____ phi_____ frames

- 57.0245 57.0000 150.0000 26

#__ type_ start__ end____ width___ exp.time_

13 omega 106.00 141.00 1.0000 1.0000

omega____ theta____ kappa____ phi_____ frames

- 57.0245 125.0000 60.0000 35

#__ type_ start__ end____ width___ exp.time_

14 omega 104.00 141.00 1.0000 1.0000

omega____ theta____ kappa____ phi_____ frames

- 57.0245 125.0000 30.0000 37

#__ type_ start__ end____ width___ exp.time_

15 omega 106.00 132.00 1.0000 1.0000

omega____ theta____ kappa____ phi_____ frames

- 57.0245 125.0000 -30.0000 26

#__ type_ start__ end____ width___ exp.time_

16 omega 77.00 106.00 1.0000 8.0000

omega____ theta____ kappa____ phi_____ frames

- 114.0489 61.0000 60.0000 29

#__ type_ start__ end____ width___ exp.time_

17 omega 103.00 178.00 1.0000 8.0000

omega____ theta____ kappa____ phi_____ frames

- 114.0489 77.0000 30.0000 75

#__ type_ start__ end____ width___ exp.time_

18 omega 113.00 175.00 1.0000 8.0000

omega____ theta____ kappa____ phi_____ frames

- 114.0489 77.0000 60.0000 62

#__ type_ start__ end____ width___ exp.time_

19 omega 108.00 173.00 1.0000 8.0000

omega____ theta____ kappa____ phi_____ frames

- 114.0489 77.0000 90.0000 65

#__ type_ start__ end____ width___ exp.time_

20 omega 91.00 172.00 1.0000 8.0000

omega____ theta____ kappa____ phi_____ frames

- 114.0489 61.0000 0.0000 81

#__ type_ start__ end____ width___ exp.time_

21 omega 102.00 127.00 1.0000 1.0000

omega____ theta____ kappa____ phi_____ frames

- 57.0245 84.0000 -169.0000 25

#__ type_ start__ end____ width___ exp.time_

22 omega 101.00 177.00 1.0000 8.0000

omega____ theta____ kappa____ phi_____ frames

- 114.0489 111.0000 -180.0000 76

#__ type_ start__ end____ width___ exp.time_

23 omega 87.00 157.00 1.0000 8.0000

omega____ theta____ kappa____ phi_____ frames

- 114.0489 61.0000 -120.0000 70

#__ type_ start__ end____ width___ exp.time_

24 omega 79.00 106.00 1.0000 8.0000

omega____ theta____ kappa____ phi_____ frames

- 114.0489 61.0000 30.0000 27

#__ type_ start__ end____ width___ exp.time_

25 omega 77.00 107.00 1.0000 8.0000

omega____ theta____ kappa____ phi_____ frames

- 114.0489 61.0000 90.0000 30

#__ type_ start__ end____ width___ exp.time_

26 omega 79.00 105.00 1.0000 8.0000

omega____ theta____ kappa____ phi_____ frames

- 114.0489 45.0000 90.0000 26

#__ type_ start__ end____ width___ exp.time_

27 omega 77.00 103.00 1.0000 8.0000

omega____ theta____ kappa____ phi_____ frames

- 114.0489 61.0000 120.0000 26

#__ type_ start__ end____ width___ exp.time_

28 omega 75.00 101.00 1.0000 8.0000

omega____ theta____ kappa____ phi_____ frames

- 114.0489 77.0000 60.0000 26

#__ type_ start__ end____ width___ exp.time_

29 omega 75.00 101.00 1.0000 8.0000

omega____ theta____ kappa____ phi_____ frames

- 114.0489 77.0000 30.0000 26

#__ type_ start__ end____ width___ exp.time_

30 omega 75.00 101.00 1.0000 8.0000

omega____ theta____ kappa____ phi_____ frames

- 114.0489 77.0000 90.0000 26

#__ type_ start__ end____ width___ exp.time_

31 omega 28.00 54.00 1.0000 8.0000

omega____ theta____ kappa____ phi_____ frames

- 114.0489 -30.0000 30.0000 26

#__ type_ start__ end____ width___ exp.time_

32 omega 80.00 153.00 1.0000 8.0000

omega____ theta____ kappa____ phi_____ frames

- 114.0489 45.0000 -60.0000 73

#__ type_ start__ end____ width___ exp.time_

33 omega 39.00 64.00 1.0000 8.0000

omega____ theta____ kappa____ phi_____ frames

- 114.0489 -45.0000 150.0000 25

#__ type_ start__ end____ width___ exp.time_

34 omega 21.00 47.00 1.0000 1.0000

omega____ theta____ kappa____ phi_____ frames

- 57.0245 57.0000 150.0000 26

#__ type_ start__ end____ width___ exp.time_

35 omega -94.00 -57.00 1.0000 1.0000

omega____ theta____ kappa____ phi_____ frames

- 0.0000 178.0000 30.0000 37

#__ type_ start__ end____ width___ exp.time_

36 omega -95.00 -35.00 1.0000 1.0000

omega____ theta____ kappa____ phi_____ frames

- 0.0000 178.0000 -180.0000 60

#__ type_ start__ end____ width___ exp.time_

37 omega -152.00 29.00 1.0000 1.0000

```
omega____ theta____ kappa____ phi______ frames
      -          -57.0245       0.0000       0.0000 181


#__ type_ start__ end____ width___ exp.time_
  38 omega -146.00    -83.00      1.0000       1.0000
omega____ theta____ kappa____ phi______ frames
      -          -57.0245    -99.0000    -60.0000 63
;
_diffrn_measurement_device              'four-circle diffractometer'
_diffrn_measurement_device_type     'SuperNova, Dual, Cu at zero, AtlasS2'
_diffrn_measurement_method              '¥w scans'
_diffrn_orient_matrix_UB_11             -0.0658741000
_diffrn_orient_matrix_UB_12             -0.0074256000
_diffrn_orient_matrix_UB_13             0.0508173000
_diffrn_orient_matrix_UB_21             -0.0577966000
_diffrn_orient_matrix_UB_22             -0.1054313000
_diffrn_orient_matrix_UB_23             -0.0604845000
_diffrn_orient_matrix_UB_31             0.0473146000
_diffrn_orient_matrix_UB_32             -0.1391212000
```

_diffrn_orient_matrix_UB_33 0.0431602000

_diffrn_radiation_monochromator mirror

_diffrn_radiation_probe x-ray

_diffrn_radiation_type CuK¥a

_diffrn_radiation_wavelength 1.54184

_diffrn_source 'sealed X-ray tube'

_diffrn_source_type 'SuperNova (Cu) X-ray Source'

_reflns_Friedel_coverage 0.000

_reflns_Friedel_fraction_full .

_reflns_Friedel_fraction_max .

_reflns_number_gt 4252

_reflns_number_total 4845

_reflns_odcompleteness_completeness 99.79

_reflns_odcompleteness_iscentric 1

_reflns_odcompleteness_theta 68.13

_reflns_special_details

;

Reflections were merged by SHELXL according to the crystal

class for the calculation of statistics and refinement.

_reflns_Friedel_fraction is defined as the number of unique

Friedel pairs measured divided by the number that would be

possible theoretically, ignoring centric projections and

systematic absences.

;

_reflns_threshold_expression 'I > 2¥s(I)'

_computing_cell_refinement

;

CrysAlisPro, Agilent Technologies,

Version 1.171.37.35 (release 13-08-2014 CrysAlis171 .NET)

(compiled Aug 13 2014,18:06:01)

;

_computing_data_collection

;

CrysAlisPro, Agilent Technologies,

Version 1.171.37.35 (release 13-08-2014 CrysAlis171 .NET)

(compiled Aug 13 2014,18:06:01)

;

_computing_data_reduction

;

CrysAlisPro, Agilent Technologies,

Version 1.171.37.35 (release 13-08-2014 CrysAlis171 .NET)

(compiled Aug 13 2014,18:06:01)

;

_computing_molecular_graphics 'Olex2 (Dolomanov et al., 2009)'

_computing_publication_material 'Olex2 (Dolomanov et al., 2009)'

_computing_structure_refinement 'ShelXL (Sheldrick, 2015)'

_computing_structure_solution 'ShelXS (Sheldrick, 2008)'

_refine_diff_density_max 0.228

_refine_diff_density_min -0.217

_refine_diff_density_rms 0.037

_refine_ls_extinction_coef .

_refine_ls_extinction_method none

_refine_ls_goodness_of_fit_ref 1.059

_refine_ls_hydrogen_treatment constr

_refine_ls_matrix_type full

_refine_ls_number_parameters 289

_refine_ls_number_reflns 4845

_refine_ls_number_restraints 36

_refine_ls_R_factor_all 0.0460

_refine_ls_R_factor_gt 0.0409

_refine_ls_restrained_S_all 1.064

_refine_ls_shift/su_max 0.000

_refine_ls_shift/su_mean 0.000

_refine_ls_structure_factor_coef Fsqd

_refine_ls_weighting_details

'w=1/[¥s^2^(Fo^2^)+(0.0650P)^2^+0.3999P] where P=(Fo^2^+2Fc^2^)/3'

_refine_ls_weighting_scheme calc

_refine_ls_wR_factor_gt 0.1147

_refine_ls_wR_factor_ref 0.1208

_refine_special_details ?

_olex2_refinement_description

;

1. Fixed Uiso

At 1.2 times of:

All C(H) groups, All C(H,H) groups

At 1.5 times of:

All C(H,H,H) groups, All O(H) groups

2. Uiso/Uaniso restraints and constraints

C20 ¥¥sim C21 ¥¥sim C22 ¥¥sim C23: within 1.7A with sigma of 0.02 and sigma for terminal atoms of 0.04

3. Others

Sof(C20)=Sof(H20A)=Sof(H20B)=Sof(C21)=Sof(H21A)=Sof(H21B)=Sof(H21C)=1-FVAR(1)

Sof(C22)=Sof(H22A)=Sof(H22B)=Sof(C23)=Sof(H23A)=Sof(H23B)=Sof(H23C)=FVAR(1)

4.a Ternary CH refined with riding coordinates:

C0AA(H0AA), C13(H13)

4.b Secondary CH2 refined with riding coordinates:

C18(H18A,H18B), C20(H20A,H20B), C22(H22A,H22B)

4.c Aromatic/amide H refined with riding coordinates:

C3(H3A), C4(H4), C5(H5), C6(H6), C7(H7)

4.d Idealised Me refined with riding coordinates:

C21(H21A,H21B,H21C), C23(H23A,H23B,H23C)

4.e Idealised Me refined as rotating group:

C8(H8A,H8B,H8C), C10(H10A,H10B,H10C), C11(H11A,H11B,H11C), C15(H15A,H15B, H15C), C16(H16A,H16B,H16C), C17(H17A,H17B,H17C), C19(H19A,H19B,H19C)

4.f Idealised tetrahedral OH refined as rotating group:

O3(H3)

;

_atom_sites_solution_hydrogens geom

_atom_sites_solution_primary ?

_atom_sites_solution_secondary ?

loop_

_atom_site_label

_atom_site_type_symbol

_atom_site_fract_x

_atom_site_fract_y

_atom_site_fract_z

_atom_site_U_iso_or_equiv

_atom_site_adp_type

_atom_site_occupancy

_atom_site_site_symmetry_order

_atom_site_calc_flag

_atom_site_refinement_flags_posn

_atom_site_refinement_flags_adp

_atom_site_refinement_flags_occupancy

_atom_site_disorder_assembly

_atom_site_disorder_group

P1 P 0.46148(3) 0.79449(4) 0.12902(2) 0.04802(13) Uani 1 1 d . . . . .

O1 O 0.42816(6) 0.77807(10) 0.27577(5) 0.0414(2) Uani 1 1 d . . . . .

O2 O 0.44215(8) 0.43577(14) 0.41253(6) 0.0592(3) Uani 1 1 d . . . . .

O3 O 0.54851(8) 0.60409(13) 0.43544(6) 0.0557(3) Uani 1 1 d . . . . .

H3 H 0.5497 0.5831 0.4810 0.083 Uiso 1 1 calc GR . . . .

O4 O 0.44396(10) 0.95772(14) 0.12429(7) 0.0681(3) Uani 1 1 d . . . . .

O5 O 0.37897(8) 0.69234(14) 0.11688(6) 0.0580(3) Uani 1 1 d . . . . .

O6 O 0.50495(10) 0.72981(16) 0.06417(7) 0.0674(3) Uani 1 1 d . . . . .

N1 N 0.50743(7) 0.69278(12) 0.28500(6) 0.0378(2) Uani 1 1 d . . . . .

C0AA C 0.42886(10) 0.87799(15) 0.34112(8) 0.0460(3) Uani 1 1 d . . . . .

H0AA H 0.4881 0.8894 0.3730 0.055 Uiso 1 1 calc R . . . .

C1AA C 0.37159(10) 0.81750(16) 0.39114(8) 0.0457(3) Uani 1 1 d . . . . .

C3 C 0.39635(13) 0.8309(2) 0.47174(10) 0.0646(4) Uani 1 1 d . . . . .

H3A H 0.4502 0.8713 0.4956 0.078 Uiso 1 1 calc R . . . .

C4 C 0.34121(19) 0.7843(3) 0.51690(12) 0.0866(7) Uani 1 1 d . . . . .

H4 H 0.3579 0.7957 0.5709 0.104 Uiso 1 1 calc R . . . .

C5 C 0.26215(18) 0.7215(3) 0.48273(15) 0.0875(7) Uani 1 1 d . . . . .

H5 H 0.2258 0.6894 0.5135 0.105 Uiso 1 1 calc R . . . .

C6 C 0.23707(15) 0.7063(3) 0.40273(14) 0.0773(6) Uani 1 1 d . . . . .

H6 H 0.1839 0.6630 0.3792 0.093 Uiso 1 1 calc R . . . .

C7 C 0.29118(12) 0.7556(2) 0.35764(10) 0.0586(4) Uani 1 1 d . . . . .

H7 H 0.2733 0.7473 0.3035 0.070 Uiso 1 1 calc R . . . .

C8 C 0.39525(16) 1.03069(19) 0.30588(13) 0.0723(5) Uani 1 1 d . . . . .

H8A H 0.4302 1.0651 0.2720 0.108 Uiso 1 1 calc GR . . . .

H8B H 0.3980 1.1032 0.3470 0.108 Uiso 1 1 calc GR . . . .

H8C H 0.3364 1.0199 0.2764 0.108 Uiso 1 1 calc GR . . . .

C9 C 0.48904(10) 0.53234(15) 0.30199(7) 0.0449(3) Uani 1 1 d . . . . .

C10 C 0.56392(15) 0.4269(2) 0.29583(10) 0.0706(5) Uani 1 1 d . . . . .

H10A H 0.5667 0.4197 0.2422 0.106 Uiso 1 1 calc GR . . . .

H10B H 0.5544 0.3279 0.3148 0.106 Uiso 1 1 calc GR . . . .

H10C H 0.6173 0.4673 0.3266 0.106 Uiso 1 1 calc GR . . . .

C11 C 0.40443(14) 0.46741(18) 0.25305(9) 0.0648(5) Uani 1 1 d . . . . .

H11A H 0.3570 0.5288 0.2597 0.097 Uiso 1 1 calc GR . . . .

H11B H 0.3972 0.3656 0.2697 0.097 Uiso 1 1 calc GR . . . .

H11C H 0.4057 0.4670 0.1989 0.097 Uiso 1 1 calc GR . . . .

C12 C 0.49037(9) 0.52358(15) 0.38946(7) 0.0419(3) Uani 1 1 d . . . . .

C13 C 0.54153(9) 0.72388(15) 0.21558(8) 0.0409(3) Uani 1 1 d . . . . .

H13 H 0.5607 0.6256 0.2001 0.049 Uiso 1 1 calc R . . . .

C14 C 0.62514(11) 0.8248(2) 0.23845(10) 0.0581(4) Uani 1 1 d . . . . .

C15 C 0.69182(13) 0.7434(3) 0.30294(16) 0.0892(7) Uani 1 1 d . . . . .

H15A H 0.6689 0.7309 0.3480 0.134 Uiso 1 1 calc GR . . . .

H15B H 0.7438 0.8026 0.3167 0.134 Uiso 1 1 calc GR . . . .

H15C H 0.7045 0.6457 0.2844 0.134 Uiso 1 1 calc GR . . . .

C16 C 0.66512(15) 0.8448(3) 0.16808(14) 0.0868(7) Uani 1 1 d . . . . .

H16A H 0.6714 0.7474 0.1458 0.130 Uiso 1 1 calc GR . . . .

H16B H 0.7208 0.8920 0.1850 0.130 Uiso 1 1 calc GR . . . .

H16C H 0.6281 0.9075 0.1295 0.130 Uiso 1 1 calc GR . . . .

C17 C 0.60793(14) 0.9813(2) 0.26869(14) 0.0773(6) Uani 1 1 d . . . . .

H17A H 0.5659 1.0336 0.2291 0.116 Uiso 1 1 calc GR . . . .

H17B H 0.6607 1.0385 0.2813 0.116 Uiso 1 1 calc GR . . . .

H17C H 0.5864 0.9700 0.3146 0.116 Uiso 1 1 calc GR . . . .

C18 C 0.29377(13) 0.7498(3) 0.11507(14) 0.0818(6) Uani 1 1 d . . . . .

H18A H 0.2725 0.7025 0.1564 0.098 Uiso 1 1 calc R . . . .

H18B H 0.2970 0.8583 0.1245 0.098 Uiso 1 1 calc R . . . .

C19 C 0.2348(2) 0.7198(5) 0.0410(2) 0.1435(15) Uani 1 1 d . . . . .

H19A H 0.2498 0.7813 0.0014 0.215 Uiso 1 1 calc GR . . . .

H19B H 0.1770 0.7434 0.0441 0.215 Uiso 1 1 calc GR . . . .

H19C H 0.2381 0.6147 0.0280 0.215 Uiso 1 1 calc GR . . . .

C20 C 0.4554(11) 0.7590(19) -0.0189(8) 0.108(4) Uani 0.412(14) 1 d . U P A -1

H20A H 0.3996 0.7979 -0.0153 0.129 Uiso 0.412(14) 1 calc R . P A -1

H20B H 0.4848 0.8428 -0.0370 0.129 Uiso 0.412(14) 1 calc R . P A -1

C21 C 0.4411(12) 0.6750(13) -0.0682(5) 0.131(4) Uani 0.412(14) 1 d . U P A -1

H21A H 0.4005 0.7181 -0.1122 0.197 Uiso 0.412(14) 1 calc R . P A -1

H21B H 0.4172 0.5845 -0.0518 0.197 Uiso 0.412(14) 1 calc R . P A -1

H21C H 0.4937 0.6505 -0.0829 0.197 Uiso 0.412(14) 1 calc R . P A -1

C22 C 0.4881(8) 0.7893(10) -0.0130(6) 0.101(3) Uani 0.588(14) 1 d . U P A 1

H22A H 0.4278 0.8191 -0.0290 0.121 Uiso 0.588(14) 1 calc R . P A 1

H22B H 0.5227 0.8799 -0.0125 0.121 Uiso 0.588(14) 1 calc R . P A 1

C23 C 0.5052(8) 0.6898(7) -0.0681(3) 0.110(3) Uani 0.588(14) 1 d . U P A 1

H23A H 0.4717 0.7180 -0.1191 0.164 Uiso 0.588(14) 1 calc R . P A 1

H23B H 0.4901 0.5886 -0.0562 0.164 Uiso 0.588(14) 1 calc R . P A 1

H23C H 0.5656 0.6936 -0.0671 0.164 Uiso 0.588(14) 1 calc R . P A 1

loop_

_atom_site_aniso_label

_atom_site_aniso_U_11

_atom_site_aniso_U_22

_atom_site_aniso_U_33

_atom_site_aniso_U_23

_atom_site_aniso_U_13

_atom_site_aniso_U_12

P1 0.0630(2) 0.0492(2) 0.03339(19) 0.00969(13) 0.01479(15) 0.00135(15)

O1 0.0460(5) 0.0439(5) 0.0363(4) 0.0014(4) 0.0139(4) 0.0043(4)

O2 0.0769(7) 0.0628(7) 0.0385(5) 0.0070(5) 0.0150(5) -0.0183(6)

O3 0.0701(7) 0.0625(6) 0.0318(5) 0.0052(4) 0.0073(4) -0.0135(5)

O4 0.0917(9) 0.0524(7) 0.0604(7) 0.0194(5) 0.0190(6) 0.0077(6)

O5 0.0592(6) 0.0664(7) 0.0440(6) 0.0067(5) 0.0039(5) -0.0031(5)

O6 0.0921(9) 0.0803(8) 0.0360(6) 0.0055(5) 0.0280(6) 0.0021(7)

N1 0.0470(6) 0.0373(5) 0.0312(5) 0.0039(4) 0.0138(4) 0.0036(4)

C0AA 0.0571(8) 0.0393(7) 0.0462(7) -0.0030(5) 0.0216(6) -0.0002(5)

C1AA 0.0576(8) 0.0429(7) 0.0404(7) 0.0023(5) 0.0191(6) 0.0071(6)

C3 0.0758(11) 0.0755(11) 0.0428(8) 0.0004(8) 0.0149(7) 0.0087(9)

C4 0.1193(19) 0.1051(17) 0.0439(9) 0.0147(10) 0.0361(11) 0.0228(14)

C5 0.1041(17) 0.0984(16) 0.0791(14) 0.0209(12) 0.0590(14) 0.0069(13)

C6 0.0742(12) 0.0839(13) 0.0861(14) -0.0024(10) 0.0435(11) -0.0087(10)

C7 0.0596(9) 0.0702(10) 0.0507(8) -0.0022(7) 0.0226(7) -0.0025(8)

C8 0.1069(15) 0.0421(8) 0.0842(13) 0.0107(8) 0.0548(12) 0.0122(9)

C9 0.0686(9) 0.0359(6) 0.0305(6) 0.0038(5) 0.0129(6) 0.0024(6)

C10 0.1127(16) 0.0532(9) 0.0546(9) 0.0136(7) 0.0375(10) 0.0305(10)

C11 0.1010(14) 0.0462(8) 0.0385(7) 0.0035(6) 0.0005(8) -0.0220(8)

C12 0.0552(7) 0.0382(6) 0.0321(6) 0.0047(5) 0.0104(5) 0.0020(5)

C13 0.0478(7) 0.0433(6) 0.0348(6) 0.0029(5) 0.0162(5) 0.0007(5)

C14 0.0520(8) 0.0700(10) 0.0568(9) -0.0036(7) 0.0222(7) -0.0113(7)

C15 0.0504(10) 0.1213(19) 0.0875(15) 0.0042(14) 0.0007(10) -0.0083(11)

C16 0.0788(13) 0.1083(17) 0.0899(15) -0.0116(13) 0.0527(12) -0.0292(12)

C17 0.0787(12) 0.0734(12) 0.0872(14) -0.0212(10) 0.0346(11) -0.0323(10)

C18 0.0588(11) 0.1085(17) 0.0745(13) 0.0055(12) 0.0093(9) 0.0048(11)

C19 0.0797(17) 0.231(5) 0.098(2) -0.017(2) -0.0208(16) 0.026(2)

C20 0.149(8) 0.140(8) 0.036(3) 0.003(5) 0.027(5) 0.033(6)

C21 0.177(10) 0.156(7) 0.050(4) -0.013(4) 0.006(5) -0.002(7)

C22 0.188(8) 0.085(3) 0.037(3) 0.014(2) 0.040(4) -0.021(4)

C23 0.194(7) 0.100(3) 0.049(2) -0.0055(19) 0.058(3) -0.004(4)

_geom_special_details

All esds (except the esd in the dihedral angle between two l.s. planes)

are estimated using the full covariance matrix. The cell esds are taken

into account individually in the estimation of esds in distances, angles

and torsion angles; correlations between esds in cell parameters are only

used when they are defined by crystal symmetry. An approximate (isotropic)

treatment of cell esds is used for estimating esds involving l.s. planes.

loop_

_geom_bond_atom_site_label_1

_geom_bond_atom_site_label_2

_geom_bond_distance

_geom_bond_site_symmetry_2

_geom_bond_publ_flag

P1 O4 1.4648(13) . ?

P1 O5 1.5660(12) . ?

P1 O6 1.5821(12) . ?

P1 C13 1.8436(14) . ?

O1 N1 1.4460(14) . ?

O1 C0AA 1.4492(16) . ?

O2 C12 1.2287(18) . ?

O3 H3 0.8200 . ?

O3 C12 1.2865(18) . ?

O5 C18 1.444(2) . ?

O6 C20 1.508(14) . ?

O6 C22 1.423(9) . ?

N1 C9 1.4902(16) . ?

N1 C13 1.4821(15) . ?

C0AA H0AA 0.9800 . ?

C0AA C1AA 1.5139(19) . ?

C0AA C8 1.525(2) . ?

C1AA C3 1.385(2) . ?

C1AA C7 1.386(2) . ?

C3 H3A 0.9300 . ?

C3 C4 1.386(3) . ?

C4 H4 0.9300 . ?

C4 C5 1.375(4) . ?

C5 H5 0.9300 . ?

C5 C6 1.376(4) . ?

C6 H6 0.9300 . ?

C6 C7 1.380(2) . ?

C7 H7 0.9300 . ?

C8 H8A 0.9600 . ?

C8 H8B 0.9600 . ?

C8 H8C 0.9600 . ?

C9 C10 1.538(2) . ?

C9 C11 1.525(2) . ?

C9 C12 1.5403(17) . ?

C10 H10A 0.9600 . ?

C10 H10B 0.9600 . ?

C10 H10C 0.9600 . ?

C11 H11A 0.9600 . ?

C11 H11B 0.9600 . ?

C11 H11C 0.9600 . ?

C13 H13 0.9800 . ?

C13 C14 1.572(2) . ?

C14 C15 1.533(3) . ?

C14 C16 1.538(2) . ?

C14 C17 1.528(3) . ?

C15 H15A 0.9600 . ?

C15 H15B 0.9600 . ?

C15 H15C 0.9600 . ?

C16 H16A 0.9600 . ?

C16 H16B 0.9600 . ?

C16 H16C 0.9600 . ?

C17 H17A 0.9600 . ?

C17 H17B 0.9600 . ?

C17 H17C 0.9600 . ?

C18 H18A 0.9700 . ?

C18 H18B 0.9700 . ?

C18 C19 1.437(4) . ?

C19 H19A 0.9600 . ?

C19 H19B 0.9600 . ?

C19 H19C 0.9600 . ?

C20 H20A 0.9700 . ?

C20 H20B 0.9700 . ?

C20 C21 1.124(17) . ?

C20 C22 0.572(19) . ?

C20 C23 1.447(16) . ?

C21 H21A 0.9600 . ?

C21 H21B 0.9600 . ?

C21 H21C 0.9600 . ?

C21 C22 1.473(13) . ?

C21 C23 1.029(13) . ?

C22 H22A 0.9700 . ?

C22 H22B 0.9700 . ?

C22 C23 1.385(12) . ?

C23 H23A 0.9600 . ?

C23 H23B 0.9600 . ?

C23 H23C 0.9600 . ?

loop_

_geom_angle_atom_site_label_1

_geom_angle_atom_site_label_2

_geom_angle_atom_site_label_3

_geom_angle

_geom_angle_site_symmetry_1

_geom_angle_site_symmetry_3

_geom_angle_publ_flag

O4 P1 O5 114.53(8) . . ?

O4 P1 O6 115.11(7) . . ?

O4 P1 C13 117.58(7) . . ?

O5 P1 O6 101.30(7) . . ?

O5 P1 C13 107.94(6) . . ?

O6 P1 C13 98.06(7) . . ?

N1 O1 C0AA 112.95(10) . . ?

C12 O3 H3 109.5 . . ?

C18 O5 P1 123.80(14) . . ?

C20 O6 P1 115.0(6) . . ?

C22 O6 P1 123.1(5) . . ?

C22 O6 C20 22.3(7) . . ?

O1 N1 C9 107.86(10) . . ?

O1 N1 C13 107.34(9) . . ?

C13 N1 C9 118.17(10) . . ?

O1 C0AA H0AA 109.7 . . ?

O1 C0AA C1AA 111.17(11) . . ?

O1 C0AA C8 106.19(13) . . ?

C1AA C0AA H0AA 109.7 . . ?

C1AA C0AA C8 110.28(13) . . ?

C8 C0AA H0AA 109.7 . . ?

C3 C1AA C0AA 120.47(15) . . ?

C3 C1AA C7 118.27(15) . . ?

C7 C1AA C0AA 121.15(14) . . ?

C1AA C3 H3A 119.9 . . ?

C1AA C3 C4 120.2(2) . . ?

C4 C3 H3A 119.9 . . ?

C3 C4 H4 119.7 . . ?

C5 C4 C3 120.70(19) . . ?

C5 C4 H4 119.7 . . ?

C4 C5 H5 120.2 . . ?

C4 C5 C6 119.66(19) . . ?

C6 C5 H5 120.2 . . ?

C5 C6 H6 120.2 . . ?

C5 C6 C7 119.6(2) . . ?

C7 C6 H6 120.2 . . ?

C1AA C7 H7 119.3 . . ?

C6 C7 C1AA 121.49(18) . . ?

C6 C7 H7 119.3 . . ?

C0AA C8 H8A 109.5 . . ?

C0AA C8 H8B 109.5 . . ?

C0AA C8 H8C 109.5 . . ?

H8A C8 H8B 109.5 . . ?

H8A C8 H8C 109.5 . . ?

H8B C8 H8C 109.5 . . ?

N1 C9 C10 111.42(13) . . ?

N1 C9 C11 115.66(12) . . ?

N1 C9 C12 107.10(10) . . ?

C10 C9 C12 102.69(11) . . ?

C11 C9 C10 109.15(14) . . ?

C11 C9 C12 110.01(12) . . ?

C9 C10 H10A 109.5 . . ?

C9 C10 H10B 109.5 . . ?

C9 C10 H10C 109.5 . . ?

H10A C10 H10B 109.5 . . ?

H10A C10 H10C 109.5 . . ?

H10B C10 H10C 109.5 . . ?

C9 C11 H11A 109.5 . . ?

C9 C11 H11B 109.5 . . ?

C9 C11 H11C 109.5 . . ?

H11A C11 H11B 109.5 . . ?

H11A C11 H11C 109.5 . . ?

H11B C11 H11C 109.5 . . ?

O2 C12 O3 123.51(12) . . ?

O2 C12 C9 120.46(12) . . ?

O3 C12 C9 115.82(12) . . ?

P1 C13 H13 105.6 . . ?

N1 C13 P1 115.31(9) . . ?

N1 C13 H13 105.6 . . ?

N1 C13 C14 110.62(11) . . ?

C14 C13 P1 113.20(10) . . ?

C14 C13 H13 105.6 . . ?

C15 C14 C13 108.46(15) . . ?

C15 C14 C16 107.67(18) . . ?

C16 C14 C13 110.52(14) . . ?

C17 C14 C13 112.80(14) . . ?

C17 C14 C15 108.36(18) . . ?

C17 C14 C16 108.89(17) . . ?

C14 C15 H15A 109.5 . . ?

C14 C15 H15B 109.5 . . ?

C14 C15 H15C 109.5 . . ?

H15A C15 H15B 109.5 . . ?

H15A C15 H15C 109.5 . . ?

H15B C15 H15C 109.5 . . ?

C14 C16 H16A 109.5 . . ?

C14 C16 H16B 109.5 . . ?

C14 C16 H16C 109.5 . . ?

H16A C16 H16B 109.5 . . ?

H16A C16 H16C 109.5 . . ?

H16B C16 H16C 109.5 . . ?

C14 C17 H17A 109.5 . . ?

C14 C17 H17B 109.5 . . ?

C14 C17 H17C 109.5 . . ?

H17A C17 H17B 109.5 . . ?

H17A C17 H17C 109.5 . . ?

H17B C17 H17C 109.5 . . ?

O5 C18 H18A 109.4 . . ?

O5 C18 H18B 109.4 . . ?

H18A C18 H18B 108.0 . . ?

C19 C18 O5 111.1(2) . . ?

C19 C18 H18A 109.4 . . ?

C19 C18 H18B 109.4 . . ?

C18 C19 H19A 109.5 . . ?

C18 C19 H19B 109.5 . . ?

C18 C19 H19C 109.5 . . ?

H19A C19 H19B 109.5 . . ?

H19A C19 H19C 109.5 . . ?

H19B C19 H19C 109.5 . . ?

O6 C20 H20A 105.6 . . ?

O6 C20 H20B 105.6 . . ?

H20A C20 H20B 106.1 . . ?

C21 C20 O6 126.8(14) . . ?

C21 C20 H20A 105.6 . . ?

C21 C20 H20B 105.6 . . ?

C21 C20 C23 45.0(9) . . ?

C22 C20 O6 71(2) . . ?

C22 C20 H20A 129.3 . . ?

C22 C20 H20B 37.9 . . ?

C22 C20 C21 117(3) . . ?

C22 C20 C23 72(2) . . ?

C23 C20 O6 106.0(10) . . ?

C23 C20 H20A 146.6 . . ?

C23 C20 H20B 75.1 . . ?

C20 C21 H21A 109.5 . . ?

C20 C21 H21B 109.5 . . ?

C20 C21 H21C 109.5 . . ?

C20 C21 C22 20.3(10) . . ?

H21A C21 H21B 109.5 . . ?

H21A C21 H21C 109.5 . . ?

H21B C21 H21C 109.5 . . ?

C22 C21 H21A 113.4 . . ?

C22 C21 H21B 123.0 . . ?

C22 C21 H21C 89.8 . . ?

C23 C21 C20 84.4(11) . . ?

C23 C21 H21A 115.3 . . ?

C23 C21 H21B 125.0 . . ?

C23 C21 H21C 25.6 . . ?

C23 C21 C22 64.4(8) . . ?

O6 C22 C21 109.1(6) . . ?

O6 C22 H22A 108.6 . . ?

O6 C22 H22B 108.6 . . ?

C20 C22 O6 87(2) . . ?

C20 C22 C21 43(2) . . ?

C20 C22 H22A 43.9 . . ?

C20 C22 H22B 151.4 . . ?

C20 C22 C23 85(2) . . ?

C21 C22 H22A 71.7 . . ?

C21 C22 H22B 140.0 . . ?

H22A C22 H22B 107.6 . . ?

C23 C22 O6 114.5(7) . . ?

C23 C22 C21 42.1(6) . . ?

C23 C22 H22A 108.6 . . ?

C23 C22 H22B 108.6 . . ?

C20 C23 H23A 101.1 . . ?

C20 C23 H23B 93.3 . . ?

C20 C23 H23C 132.0 . . ?

C21 C23 C20 50.6(8) . . ?

C21 C23 C22 73.6(8) . . ?

C21 C23 H23A 72.9 . . ?

C21 C23 H23B 65.2 . . ?

C21 C23 H23C 174.7 . . ?

C22 C23 C20 23.2(8) . . ?

C22 C23 H23A 109.5 . . ?

C22 C23 H23B 109.5 . . ?

C22 C23 H23C 109.5 . . ?

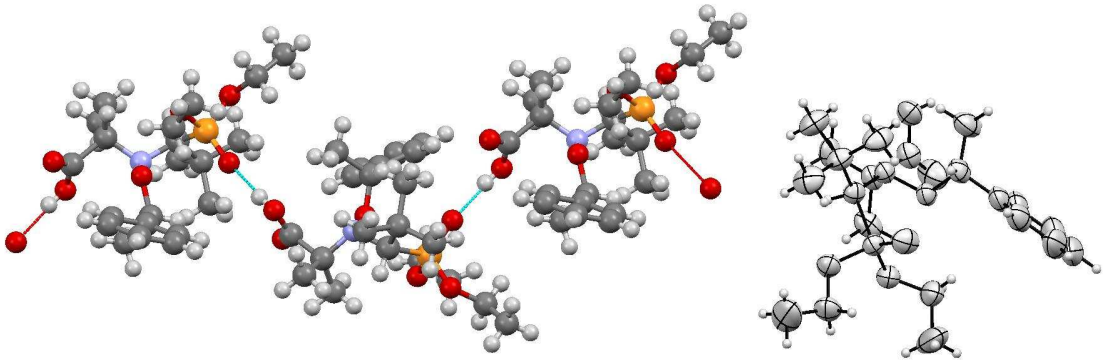
H23A C23 H23B 109.5 . . ?

H23A C23 H23C 109.5 . . ?

H23B C23 H23C 109.5 . . ?

_olex2_submission_special_instructions 'No special instructions were received'

(*RS/SR*)-4



Olex2 1.2

(compiled 2015.09.30 svn.r3233 for OlexSys, GUI svn.r5103)

;

_shelxl_version_number 2014/6

_publ_contact_author_address ?

_publ_contact_author_email ?

_publ_contact_author_name ''

_publ_contact_author_phone ?

_publ_section_references

;

Dolomanov, O.V., Bourhis, L.J., Gildea, R.J, Howard, J.A.K. & Puschmann, H.

(2009), J. Appl. Cryst. 42, 339-341.

Sheldrick, G.M. (2008). Acta Cryst. A64, 112-122.

Sheldrick, G.M. (2015). Acta Cryst. C71, 3-8.

;

_chemical_name_common ?

_chemical_name_systematic ?

_chemical_formula_moiety 'C21 H36 N O6 P'

_chemical_formula_sum 'C21 H36 N O6 P'

_chemical_formula_weight 429.48

_chemical_absolute_configuration ad

_chemical_melting_point ?

_chemical_oxdiff_formula 'c11 h36 n1 o6 p1'

loop_

_atom_type_symbol

_atom_type_description

_atom_type_scat_dispersion_real

_atom_type_scat_dispersion_imag

_atom_type_scat_source

'C' 'C' 0.0181 0.0091 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

'H' 'H' 0.0000 0.0000 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

'N' 'N' 0.0311 0.0180 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

'O' 'O' 0.0492 0.0322 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

'P' 'P' 0.2955 0.4335 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

_shelx_space_group_comment

;

The symmetry employed for this shelxl refinement is uniquely defined

by the following loop, which should always be used as a source of

symmetry information in preference to the above space-group names.

They are only intended as comments.

;

_space_group_crystal_system 'monoclinic'

_space_group_IT_number 4

_space_group_name_H-M_alt 'P 1 21 1'

_space_group_name_Hall 'P 2yb'

loop_

_space_group_symop_operation_xyz

'x, y, z'

'-x, y+1/2, -z'

_cell_length_a	8.3281(2)
_cell_length_b	15.4335(2)
_cell_length_c	10.2295(2)
_cell_angle_alpha	90
_cell_angle_beta	111.229(3)
_cell_angle_gamma	90
_cell_volume	1225.58(5)
_cell_formula_units_Z	2
_cell_measurement_reflns_used	5994
_cell_measurement_temperature	293
_cell_measurement_theta_max	74.1240
_cell_measurement_theta_min	5.4370
_shelx_estimated_absorpt_T_max	?
_shelx_estimated_absorpt_T_min	?
_exptl_absorpt_coefficient_mu	1.271
_exptl_absorpt_correction_T_max	1.00000

_exptl_absorpt_correction_T_min 0.82856

_exptl_absorpt_correction_type multi-scan

_exptl_absorpt_process_details

;

CrysAlisPro, Agilent Technologies,

Version 1.171.37.35 (release 13-08-2014 CrysAlis171 .NET)

(compiled Aug 13 2014,18:06:01)

Empirical absorption correction using spherical harmonics,

implemented in SCALE3 ABSPACK scaling algorithm.

;

_exptl_absorpt_special_details ?

_exptl_crystal_colour colourless

_exptl_crystal_colour_primary colourless

_exptl_crystal_density_diffrn 1.164

_exptl_crystal_density_meas ?

_exptl_crystal_density_method ?

_exptl_crystal_description prism

_exptl_crystal_F_000 464

_exptl_crystal_size_max 0.23

_exptl_crystal_size_mid 0.16

_exptl_crystal_size_min 0.08

_exptl_transmission_factor_max ?

_exptl_transmission_factor_min ?

_diffrn_reflns_av_R_equivalents 0.0182

_diffrn_reflns_av_unetI/netI 0.0260

_diffrn_reflns_Laue_measured_fraction_full 1.000

_diffrn_reflns_Laue_measured_fraction_max 0.986

_diffrn_reflns_limit_h_max 9

_diffrn_reflns_limit_h_min -10

_diffrn_reflns_limit_k_max 19

_diffrn_reflns_limit_k_min -19

_diffrn_reflns_limit_l_max 12

_diffrn_reflns_limit_l_min -12

_diffrn_reflns_number 8644

_diffrn_reflns_point_group_measured_fraction_full 1.000

_diffrn_reflns_point_group_measured_fraction_max 0.966

_diffrn_reflns_theta_full 67.684

_diffrn_reflns_theta_max 74.274

_diffrn_reflns_theta_min 4.637

_diffrn_ambient_temperature 293

_diffrn_detector 'CCD plate'

_diffrn_detector_area_resol_mean 5.3048

_diffrn_detector_type AtlasS2

_diffrn_measured_fraction_theta_full 1.000

_diffrn_measured_fraction_theta_max 0.986

_diffrn_measurement_details

;

#__ type_ start__ end____ width___ exp.time_

1 omega -38.00 0.00 1.0000 1.0000

omega____ theta____ kappa____ phi______ frames

- -57.0245 57.0000 -60.0000 38

#__ type_ start__ end____ width___ exp.time_

2 omega -13.00 13.00 1.0000 1.0000

omega____ theta____ kappa____ phi______ frames

- -57.0245 38.0000 -150.0000 26

#__ type_ start__ end____ width___ exp.time_

3 omega 1.00 26.00 1.0000 1.0000

omega____ theta____ kappa____ phi_____ frames

- 0.0000 -99.0000 -120.0000 25

#__ type_ start__ end____ width___ exp.time_

4 omega -73.00 -35.00 1.0000 1.0000

omega____ theta____ kappa____ phi_____ frames

- 0.0000 -178.0000 -150.0000 38

#__ type_ start__ end____ width___ exp.time_

5 omega -96.00 -41.00 1.0000 1.0000

omega____ theta____ kappa____ phi_____ frames

- 0.0000 -178.0000 0.0000 55

#__ type_ start__ end____ width___ exp.time_

6 omega -88.00 -7.00 1.0000 1.0000

omega____ theta____ kappa____ phi_____ frames

- -57.0245 125.0000 -180.0000 81

#__ type_ start__ end____ width___ exp.time_

7 omega 95.00 120.00 1.0000 1.0000

omega____ theta____ kappa____ phi_____ frames

- 57.0245 125.0000 -180.0000 25

#__ type_ start__ end____ width___ exp.time_

8 omega 81.00 176.00 1.0000 10.0000

omega____ theta____ kappa____ phi_____ frames

- 114.0489 45.0000 -120.0000 95

#__ type_ start__ end____ width___ exp.time_

9 omega 145.00 174.00 1.0000 10.0000

omega____ theta____ kappa____ phi_____ frames

- 114.0489 45.0000 0.0000 29

#__ type_ start__ end____ width___ exp.time_

10 omega 106.00 134.00 1.0000 1.0000

omega____ theta____ kappa____ phi_____ frames

- 57.0245 125.0000 120.0000 28

#__ type_ start__ end____ width___ exp.time_

11 omega 94.00 119.00 1.0000 1.0000

omega____ theta____ kappa____ phi_____ frames

- 57.0245 125.0000 90.0000 25

#__ type_ start__ end____ width___ exp.time_

12 omega 88.00 114.00 1.0000 10.0000

omega____ theta____ kappa____ phi_____ frames

- 114.0489 45.0000 0.0000 26

#__ type_ start__ end____ width___ exp.time_

13 omega 102.00 175.00 1.0000 10.0000

omega____ theta____ kappa____ phi_____ frames

- 114.0489 45.0000 -30.0000 73

#__ type_ start__ end____ width___ exp.time_

14 omega 85.00 178.00 1.0000 10.0000

omega____ theta____ kappa____ phi_____ frames

- 114.0489 45.0000 -90.0000 93

#__ type_ start__ end____ width___ exp.time_

15 omega 75.00 177.00 1.0000 10.0000

omega____ theta____ kappa____ phi_____ frames

- 114.0489 77.0000 -180.0000 102

#__ type_ start__ end____ width___ exp.time_

16 omega 71.00 170.00 1.0000 10.0000

omega____ theta____ kappa____ phi_____ frames

- 114.0489 30.0000 60.0000 99

#__ type_ start__ end____ width___ exp.time_

17 omega 72.00 98.00 1.0000 10.0000

omega____ theta____ kappa____ phi_____ frames

- 114.0489 -111.0000 60.0000 26

#__ type_ start__ end____ width___ exp.time_

18 omega 49.00 107.00 1.0000 10.0000

omega____ theta____ kappa____ phi_____ frames

- 114.0489 -61.0000 30.0000 58

#__ type_ start__ end____ width___ exp.time_

19 omega 36.00 66.00 1.0000 10.0000

omega____ theta____ kappa____ phi_____ frames

- 114.0489 -111.0000 60.0000 30

#__ type_ start__ end____ width___ exp.time_

20 omega 31.00 107.00 1.0000 10.0000

omega____ theta____ kappa____ phi_____ frames

- 114.0489 -94.0000 -150.0000 76

#__ type_ start__ end____ width___ exp.time_

21 omega 44.00 94.00 1.0000 10.0000

omega____ theta____ kappa____ phi_____ frames

- 114.0489 -45.0000 -180.0000 50

#__ type_ start__ end____ width___ exp.time_

22 omega 29.00 99.00 1.0000 10.0000

omega____ theta____ kappa____ phi_____ frames

- 114.0489 -111.0000 -180.0000 70

#__ type_ start__ end____ width___ exp.time_

23 omega -93.00 -35.00 1.0000 1.0000

omega____ theta____ kappa____ phi_____ frames

- 0.0000 178.0000 150.0000 58

#__ type_ start__ end____ width___ exp.time_

24 omega -140.00 -21.00 1.0000 1.0000

omega____ theta____ kappa____ phi_____ frames

- -57.0245 -38.0000 150.0000 119

#__ type_ start__ end____ width___ exp.time_

25 omega -139.00 -22.00 1.0000 1.0000

omega____ theta____ kappa____ phi_____ frames

- -57.0245 -57.0000 -30.0000 117

#__ type_ start__ end____ width___ exp.time_

26 omega -140.00 -74.00 1.0000 1.0000

omega____ theta____ kappa____ phi______ frames

- -57.0245 -125.0000 30.0000 66

;

_diffrn_measurement_device 'four-circle diffractometer'

_diffrn_measurement_device_type 'SuperNova, Dual, Cu at zero, AtlasS2'

_diffrn_measurement_method '¥w scans'

_diffrn_orient_matrix_UB_11 0.0569341000

_diffrn_orient_matrix_UB_12 -0.0855018000

_diffrn_orient_matrix_UB_13 0.0813304000

_diffrn_orient_matrix_UB_21 -0.1900004000

_diffrn_orient_matrix_UB_22 -0.0267448000

_diffrn_orient_matrix_UB_23 -0.0402750000

_diffrn_orient_matrix_UB_31 0.0051760000

_diffrn_orient_matrix_UB_32 -0.0439901000

_diffrn_orient_matrix_UB_33 -0.1336836000

_diffrn_radiation_monochromator mirror

_diffrn_radiation_probe x-ray

_diffrn_radiation_type CuK¥a

_diffrn_radiation_wavelength 1.54184

_diffrn_source 'sealed X-ray tube'

_diffrn_source_type 'SuperNova (Cu) X-ray Source'

_reflns_Friedel_coverage 0.887

_reflns_Friedel_fraction_full 0.999

_reflns_Friedel_fraction_max 0.945

_reflns_number_gt 4670

_reflns_number_total 4832

_reflns_odcompleteness_completeness 99.96

_reflns_odcompleteness_iscentric 1

_reflns_odcompleteness_theta 68.13

_reflns_special_details

;

Reflections were merged by SHELXL according to the crystal

class for the calculation of statistics and refinement.

_reflns_Friedel_fraction is defined as the number of unique

Friedel pairs measured divided by the number that would be

possible theoretically, ignoring centric projections and

systematic absences.

;

_reflns_threshold_expression 'I > 2¥s(I)'

_computing_cell_refinement

;

CrysAlisPro, Agilent Technologies,

Version 1.171.37.35 (release 13-08-2014 CrysAlis171 .NET)

(compiled Aug 13 2014,18:06:01)

;

_computing_data_collection

;

CrysAlisPro, Agilent Technologies,

Version 1.171.37.35 (release 13-08-2014 CrysAlis171 .NET)

(compiled Aug 13 2014,18:06:01)

;

_computing_data_reduction

;

CrysAlisPro, Agilent Technologies,

Version 1.171.37.35 (release 13-08-2014 CrysAlis171 .NET)

(compiled Aug 13 2014,18:06:01)

;

_computing_molecular_graphics 'Olex2 (Dolomanov et al., 2009)'

_computing_publication_material 'Olex2 (Dolomanov et al., 2009)'

_computing_structure_refinement 'ShelXL (Sheldrick, 2015)'

_computing_structure_solution 'ShelXS (Sheldrick, 2008)'

_refine_diff_density_max 0.193

_refine_diff_density_min -0.166

_refine_diff_density_rms 0.027

_refine_ls_abs_structure_details

;

Flack x determined using 2142 quotients [(I+)-(I-)]/[(I+)+(I-)]

(Parsons, Flack and Wagner, Acta Cryst. B69 (2013) 249-259).

;

_refine_ls_abs_structure_Flack 0.028(8)

_refine_ls_extinction_coef .

_refine_ls_extinction_method none

_refine_ls_goodness_of_fit_ref 1.032

_refine_ls_hydrogen_treatment constr

_refine_ls_matrix_type full

_refine_ls_number_parameters 271

_refine_ls_number_reflns 4832

_refine_ls_number_restraints 1

_refine_ls_R_factor_all 0.0318

_refine_ls_R_factor_gt 0.0305

_refine_ls_restrained_S_all 1.032

_refine_ls_shift/su_max 0.000

_refine_ls_shift/su_mean 0.000

_refine_ls_structure_factor_coef Fsqd

_refine_ls_weighting_details

'w=1/[¥s^2^(Fo^2^)+(0.0518P)^2^+0.0573P] where P=(Fo^2^+2Fc^2^)/3'

_refine_ls_weighting_scheme calc

_refine_ls_wR_factor_gt 0.0840

_refine_ls_wR_factor_ref 0.0856

_refine_special_details ?

_olex2_refinement_description

;

1. Fixed Uiso

At 1.2 times of:

All C(H) groups, All C(H,H) groups

At 1.5 times of:

All C(H,H,H) groups, All O(H) groups

2.a Ternary CH refined with riding coordinates:

C1(H1), C13(H13)

2.b Secondary CH2 refined with riding coordinates:

C18(H18A,H18B), C20(H20A,H20B)

2.c Aromatic/amide H refined with riding coordinates:

C3(H3A), C4(H4), C5(H5), C6(H6), C7(H7)

2.d Idealised Me refined as rotating group:

C8(H8A,H8B,H8C), C10(H10A,H10B,H10C), C11(H11A,H11B,H11C), C15(H15A,H15B,

H15C), C16(H16A,H16B,H16C), C17(H17A,H17B,H17C), C19(H19A,H19B,H19C), C21(H21A,

H21B,H21C)

2.e Idealised tetrahedral OH refined as rotating group:

O3(H3)

;

```
_atom_sites_solution_hydrogens       geom
_atom_sites_solution_primary         ?
_atom_sites_solution_secondary       ?
loop_
   _atom_site_label
   _atom_site_type_symbol
   _atom_site_fract_x
   _atom_site_fract_y
   _atom_site_fract_z
   _atom_site_U_iso_or_equiv
   _atom_site_adp_type
   _atom_site_occupancy
   _atom_site_site_symmetry_order
   _atom_site_calc_flag
   _atom_site_refinement_flags_posn
   _atom_site_refinement_flags_adp
   _atom_site_refinement_flags_occupancy
   _atom_site_disorder_assembly
   _atom_site_disorder_group
```

P1 P 0.20036(6) 0.62070(3) 0.88479(5) 0.04735(13) Uani 1 1 d . . . . .

O1 O 0.1101(2) 0.76786(10) 0.99336(16) 0.0506(3) Uani 1 1 d . . . . .

O2 O 0.2092(3) 0.96370(17) 1.0070(3) 0.0896(7) Uani 1 1 d . . . . .

O3 O -0.0608(3) 0.95946(13) 0.8601(2) 0.0703(5) Uani 1 1 d . . . . .

H3 H -0.0746 0.9954 0.9141 0.106 Uiso 1 1 calc GR . . . .

O4 O 0.1287(3) 0.56794(13) 0.9697(2) 0.0690(5) Uani 1 1 d . . . . .

O5 O 0.3779(2) 0.66309(12) 0.97216(17) 0.0569(4) Uani 1 1 d . . . . .

O6 O 0.2493(2) 0.56923(11) 0.77106(18) 0.0597(4) Uani 1 1 d . . . . .

N1 N 0.0339(2) 0.78192(11) 0.84256(18) 0.0469(4) Uani 1 1 d . . . . .

C1 C -0.0141(3) 0.75100(17) 1.0602(3) 0.0588(5) Uani 1 1 d . . . . .

H1 H -0.0684 0.6946 1.0290 0.071 Uiso 1 1 calc R . . . .

C2 C 0.0975(4) 0.74583(19) 1.2146(3) 0.0638(6) Uani 1 1 d . . . . .

C3 C 0.1035(5) 0.6703(2) 1.2888(4) 0.0839(9) Uani 1 1 d . . . . .

H3A H 0.0380 0.6226 1.2444 0.101 Uiso 1 1 calc R . . . .

C4 C 0.2073(7) 0.6656(4) 1.4298(5) 0.1088(16) Uani 1 1 d . . . . .

H4 H 0.2108 0.6148 1.4795 0.131 Uiso 1 1 calc R . . . .

C5 C 0.3045(7) 0.7354(5) 1.4962(4) 0.1169(19) Uani 1 1 d . . . . .

H5 H 0.3734 0.7318 1.5907 0.140 Uiso 1 1 calc R . . . .

C6 C 0.3000(6) 0.8106(4) 1.4231(4) 0.1102(15) Uani 1 1 d . . . . .

H6 H 0.3665 0.8579 1.4677 0.132 Uiso 1 1 calc R . . . .

C7 C 0.1953(5) 0.8157(3) 1.2817(3) 0.0851(9) Uani 1 1 d . . . . .

H7 H 0.1915 0.8668 1.2324 0.102 Uiso 1 1 calc R . . . .

C8 C -0.1526(5) 0.8196(2) 1.0324(4) 0.0827(9) Uani 1 1 d . . . . .

H8A H -0.1003 0.8742 1.0691 0.124 Uiso 1 1 calc GR . . . .

H8B H -0.2313 0.8034 1.0773 0.124 Uiso 1 1 calc GR . . . .

H8C H -0.2139 0.8247 0.9332 0.124 Uiso 1 1 calc GR . . . .

C9 C 0.1320(3) 0.85864(13) 0.8194(2) 0.0528(5) Uani 1 1 d . . . . .

C10 C 0.0576(4) 0.88708(16) 0.6656(3) 0.0683(7) Uani 1 1 d . . . . .

H10A H 0.0785 0.8428 0.6077 0.102 Uiso 1 1 calc GR . . . .

H10B H 0.1119 0.9400 0.6544 0.102 Uiso 1 1 calc GR . . . .

H10C H -0.0642 0.8963 0.6382 0.102 Uiso 1 1 calc GR . . . .

C11 C 0.3265(4) 0.84581(17) 0.8647(3) 0.0683(6) Uani 1 1 d . . . . .

H11A H 0.3739 0.8329 0.9631 0.103 Uiso 1 1 calc GR . . . .

H11B H 0.3782 0.8978 0.8466 0.103 Uiso 1 1 calc GR . . . .

H11C H 0.3496 0.7987 0.8128 0.103 Uiso 1 1 calc GR . . . .

C12 C 0.0992(3) 0.93283(15) 0.9078(3) 0.0579(5) Uani 1 1 d . . . . .

C13 C 0.0512(3) 0.70056(12) 0.7692(2) 0.0467(4) Uani 1 1 d . . . . .

H13 H 0.1028 0.7180 0.7011 0.056 Uiso 1 1 calc R . . . .

C14 C -0.1316(3) 0.66165(17) 0.6799(3) 0.0596(5) Uani 1 1 d . . . . .

C15 C -0.2235(4) 0.7272(2) 0.5649(3) 0.0787(8) Uani 1 1 d . . . . .

H15A H -0.2440 0.7796 0.6070 0.118 Uiso 1 1 calc GR . . . .

H15B H -0.3314 0.7035 0.5045 0.118 Uiso 1 1 calc GR . . . .

H15C H -0.1527 0.7397 0.5112 0.118 Uiso 1 1 calc GR . . . .

C16 C -0.1150(4) 0.5769(2) 0.6061(4) 0.0851(9) Uani 1 1 d . . . . .

H16A H -0.0448 0.5869 0.5510 0.128 Uiso 1 1 calc GR . . . .

H16B H -0.2273 0.5579 0.5461 0.128 Uiso 1 1 calc GR . . . .

H16C H -0.0626 0.5332 0.6751 0.128 Uiso 1 1 calc GR . . . .

C17 C -0.2437(4) 0.6441(2) 0.7663(4) 0.0775(8) Uani 1 1 d . . . . .

H17A H -0.1859 0.6042 0.8403 0.116 Uiso 1 1 calc GR . . . .

H17B H -0.3516 0.6197 0.7070 0.116 Uiso 1 1 calc GR . . . .

H17C H -0.2644 0.6975 0.8058 0.116 Uiso 1 1 calc GR . . . .

C18 C 0.4490(4) 0.6683(2) 1.1240(3) 0.0766(8) Uani 1 1 d . . . . .

H18A H 0.3635 0.6495 1.1618 0.092 Uiso 1 1 calc R . . . .

H18B H 0.4797 0.7278 1.1527 0.092 Uiso 1 1 calc R . . . .

C19 C 0.6005(7) 0.6136(4) 1.1781(4) 0.151(3) Uani 1 1 d . . . . .

H19A H 0.5660 0.5551 1.1863 0.227 Uiso 1 1 calc GR . . . .

H19B H 0.6734 0.6343 1.2686 0.227 Uiso 1 1 calc GR . . . .

H19C H 0.6624 0.6152 1.1151 0.227 Uiso 1 1 calc GR . . . .

C20 C 0.3724(4) 0.49877(18) 0.8156(3) 0.0724(7) Uani 1 1 d . . . . .

H20A H 0.3524 0.4657 0.8889 0.087 Uiso 1 1 calc R . . . .

H20B H 0.4885 0.5219 0.8532 0.087 Uiso 1 1 calc R . . . .

C21 C 0.3539(8) 0.4426(3) 0.6963(5) 0.125(2) Uani 1 1 d . . . . .

H21A H 0.2375 0.4217 0.6572 0.188 Uiso 1 1 calc GR . . . .

H21B H 0.4313 0.3944 0.7268 0.188 Uiso 1 1 calc GR . . . .

H21C H 0.3805 0.4747 0.6264 0.188 Uiso 1 1 calc GR . . . .

loop_

_atom_site_aniso_label

_atom_site_aniso_U_11

_atom_site_aniso_U_22

_atom_site_aniso_U_33

_atom_site_aniso_U_23

_atom_site_aniso_U_13

_atom_site_aniso_U_12

P1 0.0531(2) 0.0399(2) 0.0504(2) 0.0062(2) 0.02021(19) 0.0044(2)

O1 0.0560(8) 0.0544(8) 0.0473(7) 0.0039(6) 0.0258(6) 0.0044(6)

O2 0.0793(13) 0.0824(13) 0.1044(17) -0.0413(13) 0.0299(12) -0.0115(11)

O3 0.0824(12) 0.0574(9) 0.0704(11) -0.0099(8) 0.0266(9) 0.0173(9)

O4 0.0728(11) 0.0600(10) 0.0809(12) 0.0251(9) 0.0362(9) 0.0058(8)

O5 0.0526(8) 0.0626(9) 0.0551(8) 0.0000(7) 0.0191(7) 0.0045(7)

O6 0.0699(10) 0.0501(8) 0.0573(8) -0.0001(7) 0.0209(7) 0.0133(7)

N1 0.0553(9) 0.0415(8) 0.0477(8) 0.0041(6) 0.0231(7) 0.0035(6)

C1 0.0649(13) 0.0607(13) 0.0625(13) 0.0074(10) 0.0373(11) 0.0054(10)

C2 0.0776(15) 0.0697(14) 0.0591(13) 0.0070(11) 0.0427(12) 0.0150(12)

C3 0.102(2) 0.089(2) 0.0819(19) 0.0247(16) 0.0575(18) 0.0276(18)

C4 0.130(3) 0.136(4) 0.081(2) 0.045(3) 0.062(2) 0.055(3)

C5 0.108(3) 0.193(6) 0.0603(18) 0.013(3) 0.0429(19) 0.053(3)

C6 0.104(3) 0.159(4) 0.074(2) -0.029(3) 0.0390(19) 0.006(3)

C7 0.107(2) 0.092(2) 0.0677(16) -0.0035(15) 0.0450(17) 0.0083(18)

C8 0.0831(19) 0.089(2) 0.096(2) 0.0244(17) 0.0568(18) 0.0275(17)

C9 0.0686(13) 0.0399(9) 0.0583(12) 0.0024(8) 0.0331(10) 0.0008(9)

C10 0.105(2) 0.0487(12) 0.0634(14) 0.0092(10) 0.0451(14) 0.0049(12)

C11 0.0712(15) 0.0519(12) 0.0957(19) 0.0001(12) 0.0467(14) -0.0055(11)

C12 0.0719(14) 0.0420(10) 0.0659(13) -0.0020(9) 0.0321(11) -0.0035(9)

C13 0.0534(10) 0.0398(9) 0.0465(9) 0.0047(7) 0.0176(8) 0.0023(8)

C14 0.0541(11) 0.0553(12) 0.0602(12) 0.0023(10) 0.0098(10) -0.0023(9)

C15 0.0673(16) 0.0825(19) 0.0680(16) 0.0111(14) 0.0026(13) 0.0035(13)

C16 0.0759(18) 0.0692(17) 0.091(2) -0.0199(16) 0.0073(15) -0.0087(14)

C17 0.0552(12) 0.086(2) 0.0824(17) 0.0103(14) 0.0145(12) -0.0125(12)

C18 0.0710(15) 0.091(2) 0.0585(14) -0.0133(14) 0.0126(12) 0.0143(15)

C19 0.146(4) 0.188(6) 0.076(2) -0.016(3) -0.012(2) 0.100(4)

C20 0.0829(17) 0.0587(14) 0.0782(17) 0.0064(13) 0.0321(14) 0.0239(13)

C21 0.174(5) 0.095(3) 0.096(3) -0.019(2) 0.035(3) 0.062(3)

_geom_special_details

;

All esds (except the esd in the dihedral angle between two l.s. planes)

are estimated using the full covariance matrix. The cell esds are taken

into account individually in the estimation of esds in distances, angles

and torsion angles; correlations between esds in cell parameters are only

used when they are defined by crystal symmetry. An approximate (isotropic)

treatment of cell esds is used for estimating esds involving l.s. planes.

;

loop_

_geom_bond_atom_site_label_1

_geom_bond_atom_site_label_2

_geom_bond_distance

_geom_bond_site_symmetry_2

_geom_bond_publ_flag

P1 O4 1.4657(18) . ?

P1 O5 1.5672(18) . ?

P1 O6 1.5791(17) . ?

P1 C13 1.843(2) . ?

O1 N1 1.456(2) . ?

O1 C1 1.455(3) . ?

O2 C12 1.193(4) . ?

O3 H3 0.8200 . ?

O3 C12 1.308(3) . ?

O5 C18 1.450(3) . ?

O6 C20 1.450(3) . ?

N1 C9 1.505(3) . ?

N1 C13 1.496(2) . ?

C1 H1 0.9800 . ?

C1 C2 1.517(4) . ?

C1 C8 1.515(4) . ?

C2 C3 1.383(4) . ?

C2 C7 1.376(5) . ?

C3 H3A 0.9300 . ?

C3 C4 1.389(6) . ?

C4 H4 0.9300 . ?

C4 C5 1.370(8) . ?

C5 H5 0.9300 . ?

C5 C6 1.374(8) . ?

C6 H6 0.9300 . ?

C6 C7 1.395(5) . ?

C7 H7 0.9300 . ?

C8 H8A 0.9600 . ?

C8 H8B 0.9600 . ?

C8 H8C 0.9600 . ?

C9 C10 1.532(3) . ?

C9 C11 1.528(4) . ?

C9 C12 1.543(3) . ?

C10 H10A 0.9600 . ?

C10 H10B 0.9600 . ?

C10 H10C 0.9600 . ?

C11 H11A 0.9600 . ?

C11 H11B 0.9600 . ?

C11 H11C 0.9600 . ?

C13 H13 0.9800 . ?

C13 C14 1.583(3) . ?

C14 C15 1.530(4) . ?

C14 C16 1.541(4) . ?

C14 C17 1.524(4) . ?

C15 H15A 0.9600 . ?

C15 H15B 0.9600 . ?

C15 H15C 0.9600 . ?

C16 H16A 0.9600 . ?

C16 H16B 0.9600 . ?

C16 H16C 0.9600 . ?

C17 H17A 0.9600 . ?

C17 H17B 0.9600 . ?

C17 H17C 0.9600 . ?

C18 H18A 0.9700 . ?

C18 H18B 0.9700 . ?

C18 C19 1.451(5) . ?

C19 H19A 0.9600 . ?

C19 H19B 0.9600 . ?

C19 H19C 0.9600 . ?

C20 H20A 0.9700 . ?

C20 H20B 0.9700 . ?

C20 C21 1.459(5) . ?

C21 H21A 0.9600 . ?

C21 H21B 0.9600 . ?

C21 H21C 0.9600 . ?

loop_

_geom_angle_atom_site_label_1

_geom_angle_atom_site_label_2

_geom_angle_atom_site_label_3

_geom_angle

_geom_angle_site_symmetry_1

_geom_angle_site_symmetry_3

_geom_angle_publ_flag

O4 P1 O5 113.37(11) . . ?

O4 P1 O6 115.20(12) . . ?

O4 P1 C13 115.27(11) . . ?

O5 P1 O6 100.86(10) . . ?

O5 P1 C13 111.37(9) . . ?

O6 P1 C13 99.07(9) . . ?

C1 O1 N1 114.42(17) . . ?

C12 O3 H3 109.5 . . ?

C18 O5 P1 124.66(19) . . ?

C20 O6 P1 119.16(17) . . ?

O1 N1 C9 103.35(16) . . ?

O1 N1 C13 108.79(14) . . ?

C13 N1 C9 115.19(15) . . ?

O1 C1 H1 109.2 . . ?

O1 C1 C2 103.1(2) . . ?

O1 C1 C8 114.2(2) . . ?

C2 C1 H1 109.2 . . ?

C8 C1 H1 109.2 . . ?

C8 C1 C2 111.7(2) . . ?

C3 C2 C1 120.2(3) . . ?

C7 C2 C1 120.5(3) . . ?

C7 C2 C3 119.4(3) . . ?

C2 C3 H3A 120.0 . . ?

C2 C3 C4 119.9(4) . . ?

C4 C3 H3A 120.0 . . ?

C3 C4 H4 119.7 . . ?

C5 C4 C3 120.6(4) . . ?

C5 C4 H4 119.7 . . ?

C4 C5 H5 120.0 . . ?

C4 C5 C6 119.9(4) . . ?

C6 C5 H5 120.0 . . ?

C5 C6 H6 120.1 . . ?

C5 C6 C7 119.7(5) . . ?

C7 C6 H6 120.1 . . ?

C2 C7 C6 120.5(4) . . ?

C2 C7 H7 119.7 . . ?

C6 C7 H7 119.7 . . ?

C1 C8 H8A 109.5 . . ?

C1 C8 H8B 109.5 . . ?

C1 C8 H8C 109.5 . . ?

H8A C8 H8B 109.5 . . ?

H8A C8 H8C 109.5 . . ?

H8B C8 H8C 109.5 . . ?

N1 C9 C10 110.3(2) . . ?

N1 C9 C11 115.24(18) . . ?

N1 C9 C12 105.69(17) . . ?

C10 C9 C12 107.3(2) . . ?

C11 C9 C10 109.8(2) . . ?

C11 C9 C12 108.2(2) . . ?

C9 C10 H10A 109.5 . . ?

C9 C10 H10B 109.5 . . ?

C9 C10 H10C 109.5 . . ?

H10A C10 H10B 109.5 . . ?

H10A C10 H10C 109.5 . . ?

H10B C10 H10C 109.5 . . ?

C9 C11 H11A 109.5 . . ?

C9 C11 H11B 109.5 . . ?

C9 C11 H11C 109.5 . . ?

H11A C11 H11B 109.5 . . ?

H11A C11 H11C 109.5 . . ?

H11B C11 H11C 109.5 . . ?

O2 C12 O3 123.8(2) . . ?

O2 C12 C9 123.4(2) . . ?

O3 C12 C9 112.8(2) . . ?

P1 C13 H13 105.8 . . ?

N1 C13 P1 113.86(13) . . ?

N1 C13 H13 105.8 . . ?

N1 C13 C14 111.16(18) . . ?

C14 C13 P1 113.62(15) . . ?

C14 C13 H13 105.8 . . ?

C15 C14 C13 107.5(2) . . ?

C15 C14 C16 107.0(2) . . ?

C16 C14 C13 111.5(2) . . ?

C17 C14 C13 113.3(2) . . ?

C17 C14 C15 108.8(2) . . ?

C17 C14 C16 108.4(3) . . ?

C14 C15 H15A 109.5 . . ?

C14 C15 H15B 109.5 . . ?

C14 C15 H15C 109.5 . . ?

H15A C15 H15B 109.5 . . ?

H15A C15 H15C 109.5 . . ?

H15B C15 H15C 109.5 . . ?

C14 C16 H16A 109.5 . . ?

C14 C16 H16B 109.5 . . ?

C14 C16 H16C 109.5 . . ?

H16A C16 H16B 109.5 . . ?

H16A C16 H16C 109.5 . . ?

H16B C16 H16C 109.5 . . ?

C14 C17 H17A 109.5 . . ?

C14 C17 H17B 109.5 . . ?

C14 C17 H17C 109.5 . . ?

H17A C17 H17B 109.5 . . ?

H17A C17 H17C 109.5 . . ?

H17B C17 H17C 109.5 . . ?

O5 C18 H18A 109.7 . . ?

O5 C18 H18B 109.7 . . ?

O5 C18 C19 109.8(3) . . ?

H18A C18 H18B 108.2 . . ?

C19 C18 H18A 109.7 . . ?

C19 C18 H18B 109.7 . . ?

C18 C19 H19A 109.5 . . ?

C18 C19 H19B 109.5 . . ?

C18 C19 H19C 109.5 . . ?

H19A C19 H19B 109.5 . . ?

H19A C19 H19C 109.5 . . ?

H19B C19 H19C 109.5 . . ?

O6 C20 H20A 109.8 . . ?

O6 C20 H20B 109.8 . . ?

O6 C20 C21 109.5(3) . . ?

H20A C20 H20B 108.2 . . ?

C21 C20 H20A 109.8 . . ?

C21 C20 H20B 109.8 . . ?

C20 C21 H21A 109.5 . . ?

C20 C21 H21B 109.5 . . ?

C20 C21 H21C 109.5 . . ?

H21A C21 H21B 109.5 . . ?

H21A C21 H21C 109.5 . . ?

H21B C21 H21C 109.5 . . ?

_olex2_submission_special_instructions 'No special instructions were received'

5. References

1. Krężel, A.; Bal, W. *J. Inorg. Biochem.* **2004**, *98*, 161-6.